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BUREAU OF MINES
JAMES BOYD, DIRECTOR

INFORMATION CIRCULAR

GERMAN HIGH-TEMPERATURE COAL-TAR INDUSTRY

BY

E. O. RHODES

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FOREWORD

This report is one of a series written by members of the Solid Fuels Mission to Germany describing wartime developments in the mining, preparation, and utilization of coal. This mission was organized early in 1945 under the auspices of the Technical Industrial Intelligence Committee, which cooperated with a counterpart British committee. In some instances engineers from other allied countries cooperated. The Solid Fuels Mission operated under the direction of a steering committee headed by Dr. C. J. Potter, Deputy Solid Fuels Administrator. The personnel of the mission consisted of H. F. Yancey, in charge, J. W. Buch, Thomas Fraser, L. L. Newman, and L. D. Schmidt of the Bureau of Mines; H. H. Lowry, Director of the Coal Research Laboratory, Carnegie Institute of Technology; H. J. Rose, Vice-President and Director of Research, Bituminous Coal Research, Inc.; and F. H. Reed, Chief Chemist, Illinois Geological Survey.

E. O. Rhodes, Manager of Development, Tar and Chemical Division, Koppers Co., Inc., while serving on a mission for the Chemical Sub-Committee of TIIC in Europe, prepared this report on the German high-temperature coal-tar industry. Because of Mr. Rhodes' extensive experience in this field and the importance of the subject material to the coal producers and those utilizing coal-tar products, the report is issued by the Bureau of Mines.

Over 50 reports were written by the members of the Solid Fuels Mission. These, together with microfilms of supporting data obtained in the course of the investigations, are on file at the Bureau of Mines, Washington, D. C., where they are available for examination. The Office of the Publication Board in the Department of Commerce is prepared to furnish photolith copies, photostats, or microfilm reproductions of all declassified reports. A Bibliography of Scientific and Industrial Reports is issued weekly by the Department of Commerce, Office of the Publication Board, and is sold by the Superintendent of Documents, Government Printing Office, Washington, 25, D. C., on a subscription basis. An initial payment of $10 will provide for approximately 9 months, and subscribers will be notified when additional remittance is necessary. All subscriptions will begin with volume 1, No. 1, unless ordered otherwise. The bibliography covers all materials and subjects and gives instructions for obtaining the desired items.

The reports of the Solid Fuels Mission relate to the following general subjects: Mining methods in the Ruhr district of Germany with special reference to mechanical cutting and loading equipment, pneumatic packing, and Diesel mine locomotives; activities of industrial research laboratories supported by the German coal and coke industry; coal preparation in western Europe; low-temperature carbonization processes; metallurgical coke from weakly coking coals; low-ash electrode carbon from coal; new developments in the production of metallurgical coke and the utilization of gas and by-products; briquetting brown coal and bituminous coal without the use of binders; recent engineering developments in gas turbines and steam generators, with special reference to use of coal; German coal-fired heating and cooking stoves; and the commercial extraction of coal with solvents for production of synthetic liquid fuels or chemical products.

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Numerous reports on the activities of German synthetic liquid-fuels plants and the plants for the production of synthesis gas from solid fuels are available in the same office as the solid fuels reports.

It is planned to publish in this series of Bureau of Mines Information Circulars the more important reports or combination of reports prepared by the members of the Solid Fuels Mission. A similar series on synthetic liquid-fuel activities is being prepared.

A. C. FIELDNER, Chief,
Fuels and Explosives Branch.
Bureau of Mines
Washington, D. C.

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UNITED STATES DEPARTMENT OF THE INTERIOR - BUREAU OF MINES

GERMAN HIGH-TEMPERATURE COAL-TAR INDUSTRY[1]

By E. O. Rhodes[2]

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2/ Manager of Development, Tar and Chemicals Division, Koppers Co., Inc.

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INTRODUCTION

Germany's wartime coal-tar industry had two main branches, which can best be considered individually because their raw materials, operations, and distributions were almost completely separated.

One branch, old and well-established, was concerned almost entirely with processing coal tar produced by the high-temperature carbonization of caking bituminous coal. Carbonization was effected principally in byproduct-coke ovens and to less extent in vertical, horizontal, and inclined retorts. The high-temperature tar was all processed in tar-distilling plants that handled high-temperature tar almost exclusively.

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The other branch, newly established, dealt with low-temperature tar that came principally from brown coal, a small amount from noncaking bituminous coal, and very little (1.5 percent) from caking bituminous coal. Over 90 percent of the low-temperature tar was produced in Lurgi-Spülgas retorts. The remainder came from Krupp-Lurgi, Geissen, Borsig-Geissen, and Rolle retorts. Most of the low-temperature tar went directly to hydrogenation plants or was used, without processing, for fuel purposes. Only a small amount was processed in tar-distilling plants, and they handled low-temperature tar exclusively.

Based upon quantities of tar involved, the two branches were about equal in size in 1943. However, they differed so much in all other respects that they were virtually two separate industries. Therefore, this report, which deals only with matters pertaining to the processing of high-temperature coal tar and the use of products derived therefrom, is entitled The German High-Temperature Coal-Tar Industry.

PURPOSE AND SCOPE OF THE INVESTIGATION

The following report covers an investigation of the German high-temperature coal-tar industry from September 15 and January 15, 1945.

The primary purpose of the investigation was to determine to what extent the processing of high-temperature coal tar and utilization of products derived therefrom advanced in Germany in the years immediately preceding and during the last war, and to collect information concerning new or improved processes or products. Such information is summarized in the following portion of this report, designated Part I.

Of secondary importance was the collection of general information and data concerning German manufacturing procedures, equipment, and uses for high-temperature coal-tar products, even though they might not have been developed during recent years. Information of this kind is contained in the individual target reports that comprise Part II.

The quantity of high-temperature coal tar processed annually in Germany by 56 tar plants during the period 1941-1943 was approximately two and one-quarter million metric tons. About seventy thousand tons additional was used by small roofing plants for the manufacture of saturants and coatings. An appendix to this report contains a tabulation of the tar plants, with the quantities of tar processed by each in 1941, 1942, and 1943. The table also gives the status of each in November 1944. The latter information was based upon observation so far as possible, but in those cases where the status could not be checked, the most reliable information available at the time was used.

The appendix also contains a table showing the origins of the tar supplies received at the tar plants. They came from bituminous coal carbonized at (a) coal mines, in byproduct ovens, (b) steel works, in byproduct ovens, and (c) gas plants, in byproduct ovens and vertical, horizontal, and inclined retorts.

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All documents collected during the investigations have been microfilmed. An index will be included with the indexes covering documents microfilmed at Griesheim by JIOA, which will be forwarded to Washington, D. C.

To collect the information for this report, 40 targets were visited in the British, American, French, and Russian zones of occupation. They included producers and users of high-temperature coal-tar products, selling organizations, engineering companies, equipment manufacturers, and research organizations. A list of the targets visited, with the location of each, follows this statement. Although it is realized that there are other targets of each type that might have been included if time had permitted, it is reasonable to assume that the information received and the conclusions reached from the targets actually visited can be extended to the German high-temperature coal-tar industry as a whole, as they represent the major portion of it.

TARGETS VISITED

Name	Location
Gesellschaft für Teerverwertung	Duisburg-Meiderich
Do.	Castrop-Rauxel
Do.	Bochum-Gerthe
Rütgerswerke Aktiengesellschaft	Castrop-Rauxel
Do.	Berlin
Chemische Fabrik Weyl (Rutgerswerke)	Mannheim-Waldhof
Gelsenkirchener Bergswerks A. G., Carolinenglück	Bochum
Gelsenkirchener Bergswerks A. G., Pluto-Wilhelm	Wanne-Eickel
Bergbau A. G., Ewald König Ludwig	Herten
Gutehoffnungshütte, Zeche Sterkrade	Oberhausen-Sterkrade
Teerdestillation Mathias Stinnes	Essen-Karnap
Hanseatische Teerproducten Fabrik, Haltermann & Co.	Hamburg
Aktiengesellschaft Johannes Jeserich	Hamburg
Binne und Sohn	Hamburg-Pinneberg
Nord Deutsche Kohlen und Cokes Werke A. G.	Hamburg-Freihafen
Dr. F. Raschig G.m.b.H.	Ludwigshafen
Dr. F. Raschig G.m.b.H., Zweiniederlassung	Bochum-Langendreer
Teerdestillation Stuttgart	Stuttgart
Ehano Asphalt-Werke Aktiengesellschaft	Hamburg-Harbourg
Emschergenossenschaft	Essen
Lothringen Bergbau Aktiengesellschaft	Bochum-Lothringen
Verkaufvereinigung für Teererzeugnisse	Castrop-Rauxel
Mannesmannrohrenwerke, Königen Elizabeth mine	Essen-Frillendorf
Heinrich Koppers G.m.b.H.	Essen
Dr. C. Otto & Co.	Bochum-Dahlhausen
Firma Carl Still	Recklinghausen
Didier Werke A. G.	Berlin-Wilmersdorf
Schumachersche Fabrik	Bietingheim
Aktiengesellschaft der Kohlenwertstoff-Verbände	Bochum
Phenol Verband	Berlin
Verkaufsvereinigung für Teererzeugnisse	Berlin

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Name	Location
I. G. Farbenindustrie	Ludwigshafen
Do.	Uerdingen
Do.	Schkopau
Chemische Werke Albert	Wiesbaden-Biebrich
Deutsche Gasrusswerke, G.m.b.H.	Dortmund
Ruhröl, G.m.b.H.	Bottrop
Ruhrchemie	Sterkrade-Holten
Gasinstitut der Technische Hochschule	Karlsruhe
Kaiser Wilhelm Institut für Kohlenforschung	Mülheim

PART I

RECENT ADVANCES IN GERMANY IN THE PROCESSING OF HIGH-TEMPERATURE COAL TAR AND USE OF PRODUCTS OBTAINED THEREFROM

Summary

In the years immediately preceding and during the last war, Germany's need for fuel oils, especially for the navy, motor fuels for Diesel and gasoline engines, and briquette fuels for transportation and industrial heating purposes caused the old and well-established branch of the German coal-tar industry that deals with the processing of high-temperature coal tar to make an extraordinary effort to produce maximum quantities of fuel oils, oils, and pitch for the hydrogenation plants, pitch for fuel briquettes, and pitch and pitch coke for carbon electrodes. Such peacetime activities as the manufacture of creosote for wood preservation and refined tars for road construction were abandoned almost completely. Few changes in manufacturing procedures or equipment were made at the tar plants, and such changes as were made resulted largely from the increased production of fuel oils, hard pitches, and pitch coke brought about by the war.

In this summary, advances in equipment, methods, products, and uses of products that have taken place within the past 10 years in the high-temperature coal-tar industry of Germany are enumerated and discussed briefly. Most of the items mentioned are discussed in greater detail in the individual target reports, which follow this summary. Some of them are discussed in greater detail in reports submitted by other investigators. Reference is made to any such reports whenever possible.

New or Improved Tar-Plant Equipment

Tar Stills

Probably the most important improvement made in any of the German tar plants was the installation of a continuous tar still of somewhat novel design at the Duisburg Meiderich plant of Gesellschaft für Teerverwertung. Prior to the installation of that unit, this company, the largest tar-distilling organization in Germany, had used only batch stills in all of its

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plants. In fact, tar distillation in virtually all of the German plants is carried on in batch distilling equipment, and installation of the new still by Gesellschaft für Teerverwertung was more significant and noticeable on that account.

Tar stills, general. - The various types of batch stills used in Germany are illustrated in figure 1. Vertical pot type stills without columns (Teerverwertung, Meiderich) predominate. Teerverwertung, Rauxel, the largest tar plant from the standpoint of volume of tar distilled, and others, use horizontal stills (without columns) heated through two large tubes. At least one plant (Carolinenglück) uses horizontal stills like those at Rauxel, connected to offset fractionating columns. Carolinenglück and most of the other tar plants use vertical stills with offset fractionating columns for the redistillation of oils. Only one company, Rütgerswerke, uses vertical stills with columns mounted directly on top. Most of its plants are provided with installations of that kind. All the different types of batch stills mentioned above are operated under vacuum, usually about 600 to 650 mm. of mercury.

Continuous tar stills are used in only a few of the high-temperature tar-distilling plants in Germany. Installation of the Koppers atmospheric type, illustrated schematically by figure 2, are used by Nord Deutsche Kohlen und Cokes Werke, A.G., at Hamburg, and by Röchling'sche Eisen und Stahlwerke, G.m.b.H., at Völklingen, Saar. The largest installation of this type is at Mahrisch Ostrau in Tschechoslowakei. The Ludwigshafen plant of Dr. F. Raschig, G.m.b.H., uses a Raschig continuous vacuum still arranged as shown in figure 3. It is the only installation of this type in Germany.

For comparison with the above-mentioned systems, the arrangement of the new continuous vacuum still of Gesellschaft für Teerverwertung at Duisburg-Meiderich is shown schematically in figure 4.

G.f.T. continuous tar still. - In principle, the G.f.T. still is similar to other continuous stills employing pipe heaters with fractionating columns operating in series, but it differs from them principally in the use of vacuum rather than atmospheric pressure in the vaporizing and fractionating columns. Coal tar dehydrated in other equipment is heated in a pipe heater under pressure, released through a constricting cock into a cast-iron, vacuum flash chamber, where the pitch is separated from the vapors. The briquette pitch flows by gravity through a pitch-to-tar heat enchanger to a pitch receiver. The vapors pass from the flash chamber successively through three steel columns packed with Raschig rings, through a final condenser, and through two scrubbers ahead of the vacuum pump. From the three columns and final condenser are discharged, by gravity, through barometric legs, anthracene oil, "Solvay" wash oil, naphthalene oil, and carbolic oil.

In addition to the use of vacuum in the columns, other items of interest in this installation are the location of the gas burners in the floor of the pipe heater instead of in the usual position at one side, a

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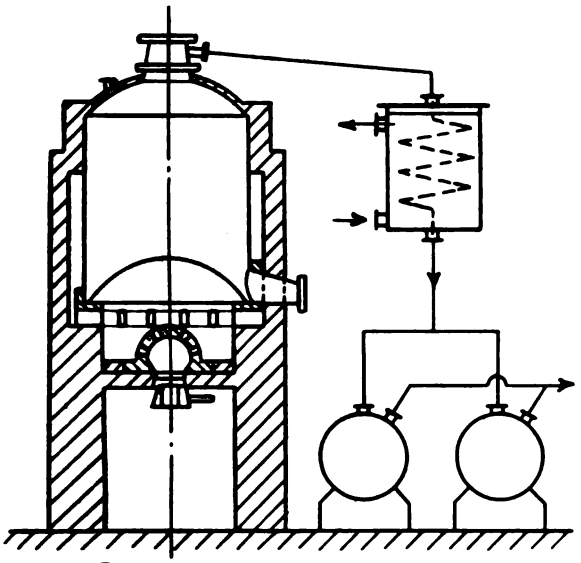
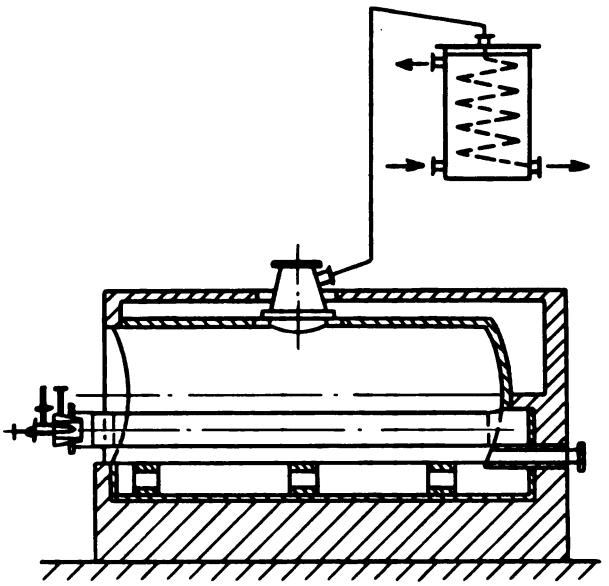
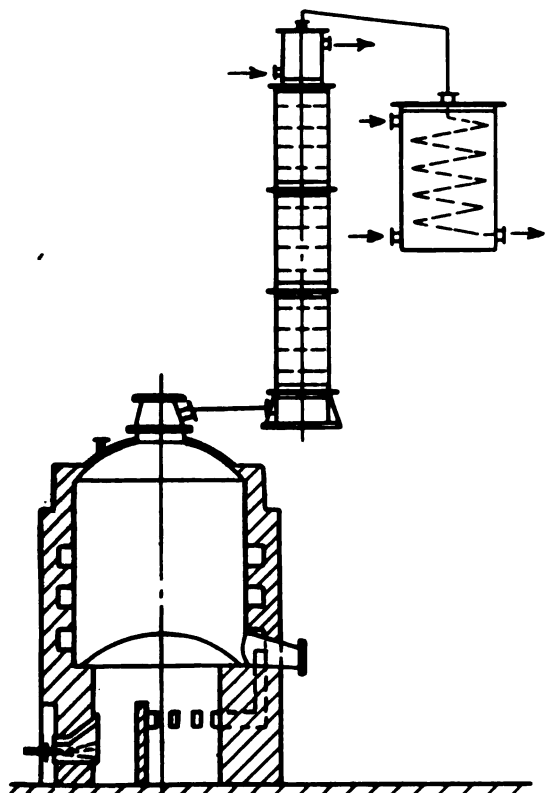


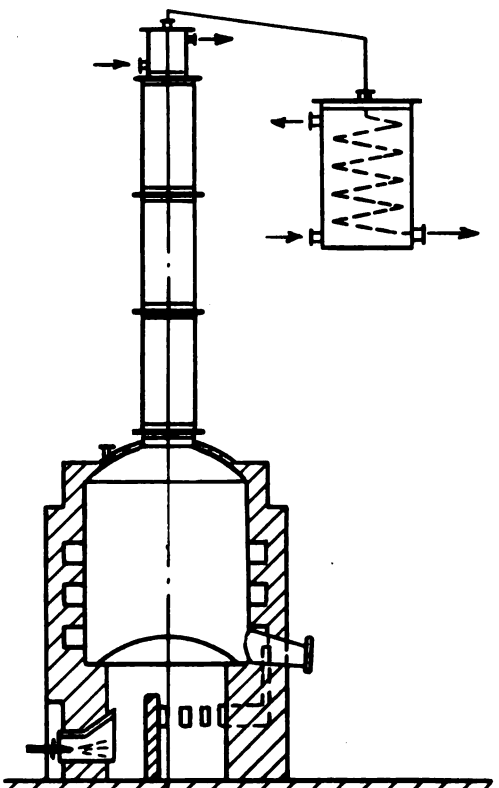
Teerverwertung, Meiderich



Teerverwertung, Rauxel



Karolinengluek, Bochum



Ruettgers, Rauxel

Figure 1. - Vacuum-batch-systems.

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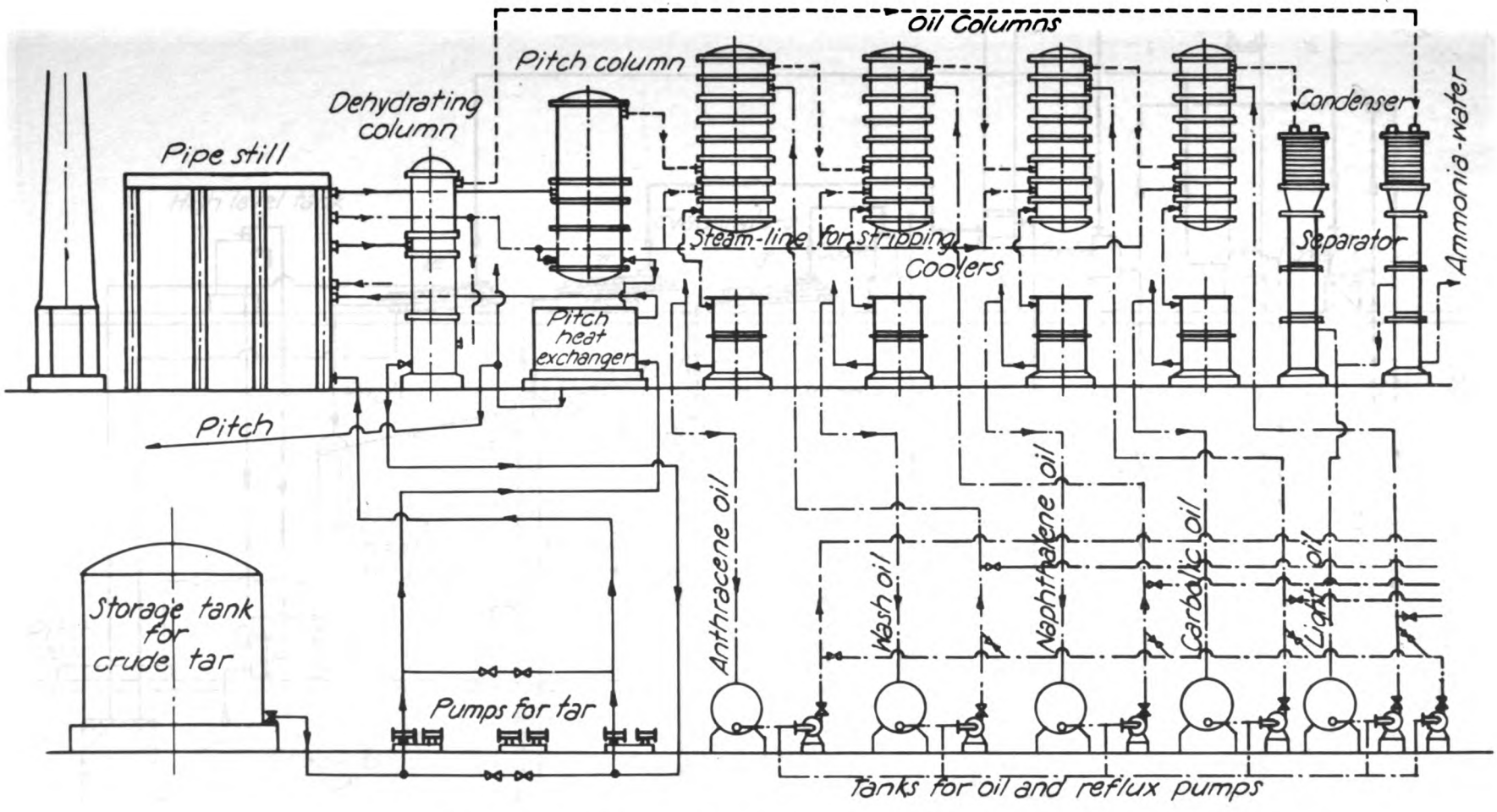

Figure 2.- Continuous Koppers atmospheric system.

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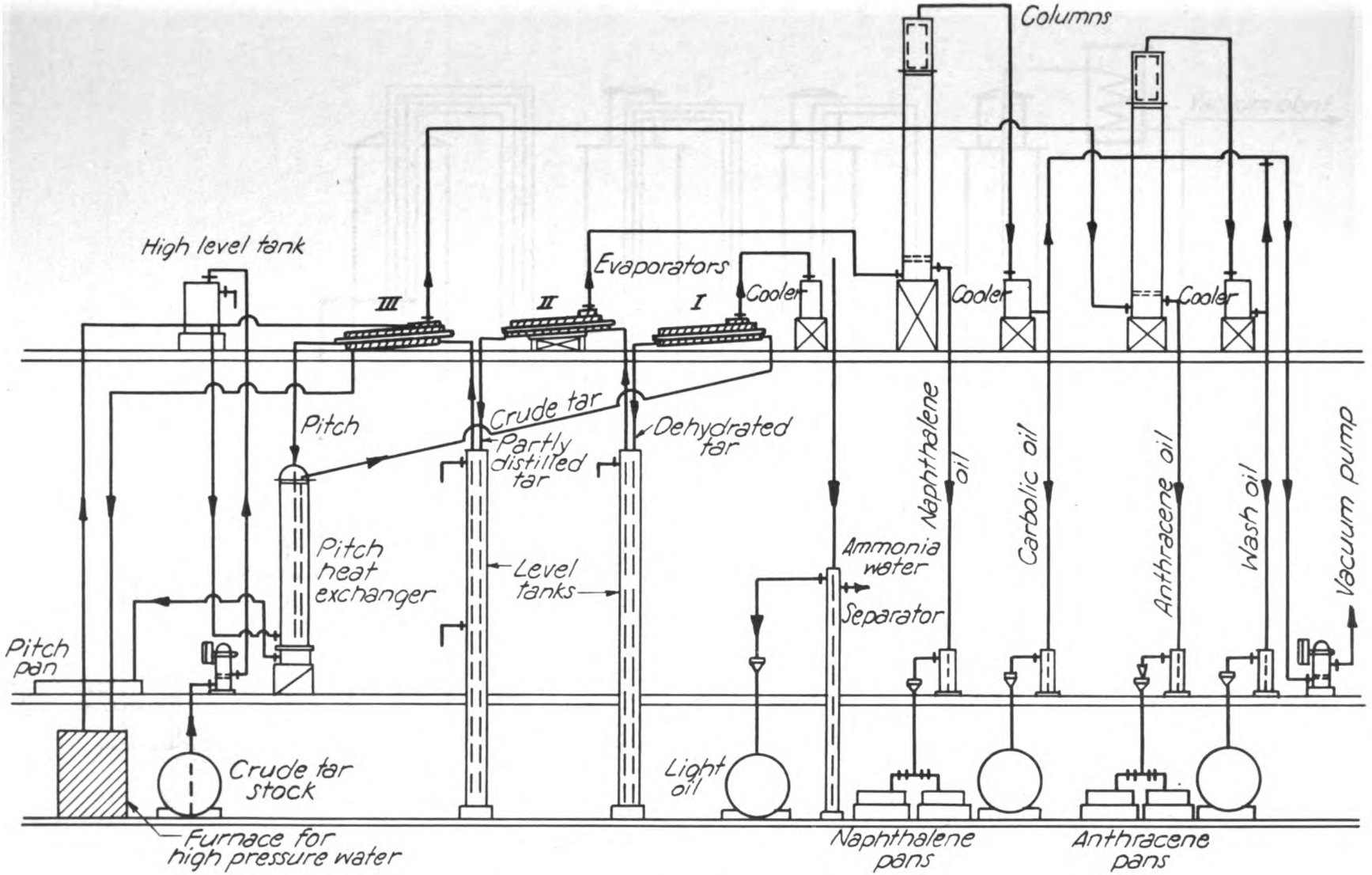

Figure 3.- Continuous Raschig vacuum system.

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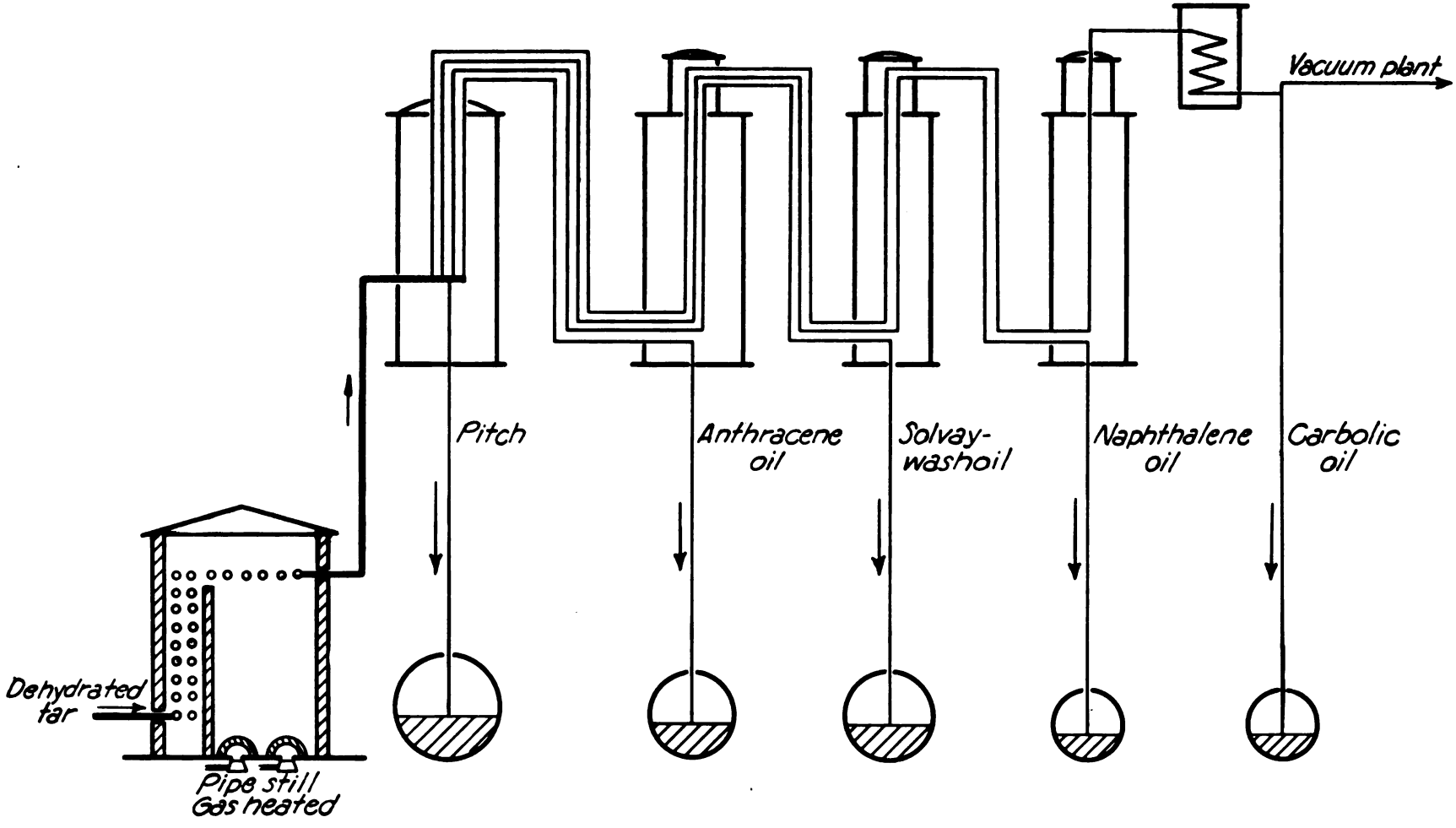

Figure 4.- Continuous Teerverwertung vacuum system.

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constricting or pressure-reducing cock between the pipe heater and the flash chamber; iron Raschig rings in the upper part of the flash chamber to protect subsequent steel parts of the equipment against corrosion, a unique method for distributing the reflux from the dephlegmator onto the packing in each column, using a distributing trough with slots in the sides to cause the reflux to fall onto the packing in two concentric and virtually continuous circles.

Some of the advantages claimed for this new installation by Gesellschaft für Teerverwertung are:

1. A heat consumption below all known values.
2. A greater yield of oil (about 3 to 5 percent).
3. Control of all corrosion influences.
4. Avoidance of coke formation in the heating tubes.

Hard-Pitch Manufacture

During recent years the use of hard pitch for pitch coke and plastic compounds created a demand for hard pitch in Germany that did not exist before to any considerable extent. To meet this demand, several of the German tar plants installed special equipment for the purpose. Others, particularly Rütgerswerke and Pluto-Wilhelm, made the hard pitch in the stills ordinarily used for the production of briquette pitch.

Gesellschaft für Teerverwertung, Duisburg-Meiderich. - At Castrop-Rauxel, Gesellschaft für Teerverwertung installed seven vertical, 50-ton stills for hard-pitch manufacture. They were gas-heated, operated under vacuum, and provided with pipes running to the bottom of each still for the introduction of air. Briquette pitch made in other stills was transferred to the hard-pitch stills, where distillation was continued under vacuum and with agitation by air drawn into the stills by vacuum. When the desired softening point of 150°C. was reached, at which time the temperature of the pitch was 380°-390°C., the pitch was dropped into transfer tanks. From these it was transferred by suction into insulated tank cars, which transported it in liquid form to the pitch coke plant.

Ewald-König Ludwig. - Bergbau A.G., Ewald-König Ludwig installed two 3-ton vertical, gas-fired stills especially for hard-pitch manufacture. Also, an older 50-ton still was sometimes used for the same purpose. Briquette pitch made in horizontal stills was transferred to the hard-pitch stills and blown with combustion gases heated by a direct flame. The combustion gases were introduced into the still under 4 to 5 atmospheres pressure through a distributing pipe. On occasions tar was distilled to briquette pitch with vacuum in the 50-ton vertical still, and then the distillation was carried to hard pitch in the same still in the manner described above.

Gesellschaft für Teerverwertung, Bochum-Gerthe. - For several years Gesellschaft für Teerverwertung at Bochum-Gerthe has used a method of

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hard-pitch manufacture similar to the one described above. Hot combustion gases entered a vertical combustion chamber, into which gas was introduced without additional air. Combustion of the gas with the oxygen contained in the combustion gas rendered the latter oxygen-free and at the same time raised its temperature to 600°C. The hot combustion gases were then drawn by suction through hot briquette pitch contained in two 50-ton stills until the temperature of the pitch was about 380°C. and softening point about 150°C. The daily capacity of each still was about 25 tons of hard pitch.

Rütgerswerke A.G., Castrop-Rauxel. - At Rauxel, Rütgerswerke A.G. made hard pitch from briquette pitch in the same stills in which the briquette pitch was produced. Continued heating with vacuum and steam but without air blowing was employed. However, Rütgerswerke has a special installation at this plant for producing extra hard pitch used for making Plastix. The installation consists of an oxidizing still of 100 tons capacity heated with gas by large ring burners under the concave bottom of the still. Both air and steam can be introduced into the still through distributors provided for that purpose. Vapors from the still are condensed by a water-cooled condenser.

A charge of 70 to 80 tons of molten briquette pitch is transferred to this equipment from the tar stills. It is then heated by the ring burners to 290°-300°C. Air blowing is started, and some steam is admitted at the same time. The quantity of air used for blowing the pitch is about 400 to 600 cubic meters per hour. It not only serves to oxidize the briquette pitch but also provides intense agitation of the entire contents of the still. Throughout the blowing period the temperature is kept at 350°C. or above by means of the ring burners. The desired softening point of 180° to 200°C. is reached in about 10 to 14 hours. The amount of distillate removed is about 5 to 10 percent. To make Plastix, the purpose of which is explained in a later portion of this summary, the high softening-point pitch is fluxed with filtered high-boiling tar oils to the desired final softening point, usually between 38° and 60°C.

Chemische Betriebe Pluto-Wilhelm. - This company made hard pitch for use in the manufacture of Plastix but did not employ special equipment for the purpose. Distillation was carried all the way to hard pitch in vertical stills with heat and vacuum only.

Pitch-Cooling Equipment

Due primarily to the production of high softening point pitch in Germany for the manufacture of pitch coke, considerable attention has been given during the past few years to the development of improved methods of cooling pitch. Special equipment was installed at some of the tar plants for this purpose.

Prior to 1938, very little hard pitch was made in Germany and the briquette pitch, the principal residual product of the tar industry, was

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cooled in open bays, in stationary steel pans usually mounted above railroad tracks, or in split molds. In at least two plants steel pans were used that could be tilted to dump the contents into cars or car-loading equipment. All of these methods were intermittent, and the tar distillers wished to use continuous methods, especially for the cooling of high softening point pitch. Various methods developed for this purpose are described in the following paragraphs.

Gelsenkirchen Mining Co., Carolinenglück plant. - One of the most interesting methods of cooling pitch was developed at the Carolinenglück plant of the Gelsenkirchen Mining Co. The equipment used in this process consisted of a horizontal tank, with valve-actuated openings on the bottom, mounted above an open rectangular tank with a hopper bottom. A screw conveyor was mounted in a trough in the lower portion of the hopper bottom of the rectangular tank. A bucket elevator with perforated buckets was mounted in an inclined position on one end of the rectangular tank. This equipment was used in the following manner: Pitch having a softening point above 160°C. was cooled in an overhead tank until its temperature was below 300°C. It was then run by gravity into the horizontal tank equipped with valve-actuated openings, and by means of the latter was dropped in small streams into water contained in the rectangular tank. The streams of pitch solidified while dropping through the water and broke into small rodlike pieces when they reached the screw conveyor. The pieces of solid pitch were then moved to the end of the tank by the screw conveyor and delivered into the bucket elevator. By means of the latter they were carried above the level of the water in the tank and discharged into a railway car. This method was said by the inventor, Mr. Georg. Speckhardt of Carolinenglück, to be satisfactory for the cooling of briquette pitch as well as hard pitch. However, when cooling briquette pitch, the temperature before entering the water should be not higher than 150°C. German patent No. 743678, covering this process, was issued to Mr. Speckhardt on May 12, 1943.

Bergbau A.G. Ewald-König Ludwig. - Another interesting pitch-cooling device was installed at the Ewald-König Ludwig plant of Bergbau A.G. at Herten near Recklinghausen. It consisted of a rotating steel drum or cylinder about 20 feet long and 2 feet in diameter. The surface of the drum was divided into shallow troughs about 4 inches wide and 1/2 inch deep by narrow ribs spaced about 4 inches apart. On one side of the drum, a short distance below the top, a water-distributing manifold sprayed water onto the surface between the ribs that comprised the side walls of the shallow troughs. A pitch-distributing manifold flowed liquid high softening-point pitch onto the films of water in the troughs. The films of pitch were cooled by the underlying films of water and by overlying air as the rotating drum carried the pitch films over the top and down the other side. While the pitch films were moving downward with the rotation of the cylinder, scrapers removed them from the drum and discharged them into sloping metal troughs, in the bottoms of which water was running. The pitch films, which were plastic when leaving the drum, were cooled further by the water in the sloping troughs and conveyed by it to a grating

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made of steel rods, set close together, which separated the water from the solidified pitch. The water fell through the grating into a tank, from which it was returned to the drum, and the solidified pitch fell onto a conveyor, which transported it to a car. The pitch arrived at the car in the form of small thin plates. This installation was said to have had a capacity of 20 tons of high softening-point pitch per hour. So far, it has been used only for cooling high softening point pitch, but tests are said to have indicated that it could be used for briquette pitch as well by making only minor changes.

G.f.T., Carolinenglück and Ruhröl G.m.b.H. - Endless, inclined conveyors were used by Gesellschaft für Teerverwertung at Rauxel and Gerthe and by the Carolinenglück tar-distilling plant for cooling high softening-point pitch. Also, the same type of device was used by Ruhröl A.G. for cooling a pitch residue (softening point 200°C.) from the distillation of filtered coal extract. These inclined conveyors were made of overlapping steel plates. The liquid pitch was flowed onto the conveyor at its lower end and solidified, with or without the aid of water sprays, as the conveyor moved to the upper end of the incline. There the pitch discharged, in the form of thin plates, into a railway car. The conveyor then returned to the lower end of the incline. In the case of Ruhröl installation, the steel conveyor passed through water to cool the conveyor plates completely before more of the molten pitch was applied.

Two conveyors of this type were used by Gesellschaft für Teerverwertung at Rauxel. The capacity of one was 50 tons (metric) per day and the capacity of the other was 200 tons per day. At the Gerthe plant of the same company, the capacity of the inclined conveyor was 50 tons per day. Pitch coolers of this type were not satisfactory for cooling briquette pitch, and when used on high softening-point pitch they were said to require excessive maintenance.

Rütgerswerke A.G., Castrop-Rauxel and Gutehoffnungshütte. - Rütgerswerke A.G. at Rauxel tried various methods of cooling pitch. One method, used only for the cooling of high softening-point pitch, consisted in flowing the liquid pitch in thin streams into water flowing in inclined shallow troughs. The streams of pitch were solidified into rods by the water and were carried to the lower ends of the troughs, where they were separated from the water by a grating made of iron bars set close together. From the grating the rods of pitch dropped into a railway car, and the water dropped through the grating into a tank, from which it was returned to the upper ends of the inclined troughs. The capacity of the Rauxel installation was said to be 170 to 240 tons of pitch per day.

Another installation of the same type was used at the Gutehoffnungshütte tar plant. At this installation the high softening-point pitch to be cooled was pre-cooled to about 250°C. It then entered a horizontal manifold 80 mm. in diameter and emerged from the bottom of the manifold through 25 nipples with 10 to 12 mm. openings.

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The streams of pitch dropped from the nipples onto water flowing in 25 troughs each 7 m. long and about 100 mm. wide. The temperature of the water was about 40°C. The water and pitch were separated at the ends of the troughs by a grating which discharged the rods of pitch into a railway car and the water into a recirculating tank. The capacity of this installation was said to be about 100 tons per day of hard pitch. The quantity of water recirculated was 25 to 30 cubic meters per hour.

Another system of pitch cooling was being developed by Rütgerswerke at Rauxel especially for the cooling of briquette pitch. Insufficient tests were made to establish its capacity or suitability for this purpose. In this system, molten briquette pitch flowed from a wide, flat nozzle onto the surface of water flowing in a slightly inclined trough 60 meters long and about 25 centimeters wide. The thin ribbon of pitch leaving the end of the nozzle floated on the surface of the water for several meters. It was cooled by the water from below and air from above to the point where no water would be occluded when the pitch became submerged. On reaching the end of the 60-meter inclined trough, the pitch and water were separated by an inclined grating, which discharged the pitch into a railway car and the water into a recirculating tank.

The two methods of cooling pitch described above were patented in Germany by Rütgerswerke A.G. in 1940 and 1943, respectively.

Anthracene Recovery Equipment

During the war most of the German tar plants recovered a product called "anthracene rückstand" or anthracene residue by the crystallization and filtration of anthracene oil. In the past it has been customary to cool the anthracene oil in shallow pans with occasional stirring by hand and then to filter the crystalline slurry through shallow vacuum filter pans. The anthracene residue was removed from the vacuum pans by hand. In some plants, centrifuges were used in place of or in addition to the vacuum filters, and sometimes plate and frame filter presses were used. In each of these cases, the anthracene residue when shipped to carbon black factories contained appreciable quantities of anthracene oil. Because the latter was in great demand for hydrogenation, fuel-oil, and fluxing purposes, and because it had a higher sales value than the anthracene residue, attempts were made to lower the oil content of the anthracene residue to a minimum. Special equipment for this purpose was installed by Gesellschaft für Teerverwertung at Meiderich in the following manner:

Gesellschaft für Teerverwertung, Meiderich. - Twenty water-cooled vertical crystallizers are mounted above five vertical basket centrifuges arranged to discharge the anthracene residue onto conveyors, which transfer it directly into open cars.

Each crystallizer is charged with 12 cubic meters of anthracene oil at 80°C. Cooling is then effected by water from the canal circulated through jackets surrounding each crystallizer. The oil is agitated continuously by a stirrer having vertical paddles close to the side walls of the crystallizers.

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The agitators are driven by overhead motors. Cooling to approximately 15°C. requires about 1-1/2 days per charge. The crystalline slurry is dropped by gravity into a centrifuge, and the cake is whizzed for 5 to 15 minutes, depending on the quality of the anthracene oil charged to the crystallizer. The baskets in the centrifuges are 2 meters in diameter. They are made of phosphor bronze. Normally, the capacity of each centrifuge is about 80 tons of slurry per 24 hours. Anthracene residue produced in this manner contains only 2 to 3 percent of anthracene oil. The anthracene content is 20 to 25 percent. The centrifuged oil remains crystal free at 0°C.

Tar-Acid Extracting Equipment

Only a few of the German tar plants are equipped for tar acid refining. During the war those plants received the crude tar acids from all of the others in the form of tar acid oils, crude tar acids, or phenolates and processed them to recover refined phenol and cresols. At most of the tar plants where tar acids were extracted, simple batch extracting equipment was used. However, at the Ludwigshafen plant of Dr. F. Raschig G.m.b.H., countercurrent extraction in a tower packed with Raschig rings, started several years before, was continued, and a new method was developed at the Carolinenglück plant of the Gelsenkirchen Mining Co.

Carolinenglück. - The method developed by Carolinenglück, patented in 1943 by Georg Speckhardt, involved the use of two cone-bottom tanks of 20 mm. capacity, each with pipes running to the bottoms of the cones and terminating in sprinkler heads with holes not larger than 10 mm. To use this equipment, each cone-bottom tank was partly filled with 20 percent caustic-soda solution. The tar-acid oil was then pumped slowly into one of the tanks through the distributor near the bottom of the cone, and the oil, after bubbling up through the caustic-soda solution, flowed out of the tank and was pumped into the second tank, where the process was repeated. When the caustic solution in the first tank became saturated with tar acids, the process was stopped and the phenolate was withdrawn from the tank and replaced by fresh caustic-soda solution. The flow of the oil was then reversed, passing first through the tank containing partly saturated caustic and then through the tank containing fresh caustic solution. If preferred, one cone-bottom tank instead of two may be used for carrying out this process.

According to Mr. Speckhardt, the use of the above-described method of removing phenols from tar acid oils tends to avoid emulsion difficulties, which are sometimes encountered when countercurrent flow of both caustic and oil is employed.

New or Increased Uses of Pitches and Tar Oils

Electrode Coke Manufacture

Lothringen Bergbau Aktiengesellschaft. - One of Germany's greatest war needs was pure carbon for electrodes used in the production of aluminum. As early as 1936, a plant had been installed at Bochum-Lothringen by Lothringen Bergbau Aktiengesellschaft to make electrode coke from hard pitch

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(150°C, K.S.). The plant consisted of 65 refractory, gas-heated, sole-flue ovens 1.5 m. wide by 10 m. long, a mechanical pusher, a quenching and cooling platform level with the oven floors, and three vertical stills for the redistillation of pitch distillate.

This plant was operated as follows: 14 tons of crushed or flaked hard pitch was charged into an oven from the top, and the pitch was distilled by heat from sole flues supplemented by combustion, inside the ovens, of gas from the pitch coke battery during the latter part of the coking cycle. It was necessary to supplement the heat from the sole flues in this manner in order to reduce the volatile matter of the coke below 1.0 percent. At the end of the coking cycle, the luted doors were raised and the coke was pushed onto the cooling and quenching platform, where it was quenched with a minimum amount of water applied by hand. It was then loaded by hand into cars.

Distillate from the ovens, amounting to about 15 percent of the charge, was redistilled to briquette pitch in vertical stills with vacuum and gas heating. The briquette pitch was then transferred to another vertical still, where it was blown with hot inert gases until a softening point of 150°C. was reached. The hard pitch so made was then added to hard pitch from the tar plants and charged into the pitch coke ovens. The distillate produced during the blowing operation went to hydrogenation plants. The inert gas used for blowing the pitch distillate to hard pitch was made by burning a small amount of coke-oven gas in hot stack gases from the pitch coke plant. This was done in a small vertical combustion chamber located beside the blowing stills.

The capacity of the Lothringen pitch coke plant was about 200 tons of pitch coke per day. It operated throughout the war and suffered only minor damage from air raids.

Verkaufsvereinigung für Teererzeugnisse, Rauxel. - In 1939, a new pitch coke plant was built to supplement the output of The Lothringen plant. The new plant was erected for Verkaufsvereinigung für Teererzeugnisse at Castrop-Rauxel near the tar-distilling plant of Gesellschaft für Teerverwertung. The latter concern operated the pitch coke plant and supplied much of the hard pitch used in its operation. The hard pitch was made by blowing briquette pitch with air in the manner previously described.

The Rauxel pitch coke plant consisted of 20 silica-lined, Koppers, regenerative, byproduct ovens separated into four blocks of five ovens each by concrete pinion walls. The ovens were 11,800 mm. long, 3,000 mm. high, and 450 mm. wide (average width). The ovens were charged with liquid, high softening-point pitch (150°C.) from six gas-heated, vertical charging tanks with internal circulating pumps. One charging tank was used for each block of five ovens, and two were held as spares. The liquid pitch was circulated by the pumps in the charging tanks through pipe loops running over the ovens and back to the charging tanks. To charge an oven, pitch flowed from the circulating loop through a nozzle inserted through one of the charging-hole covers.

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Three hours were required to charge an oven with 9 (metric) tons of pitch. During a part of this charging period a mixture of preheated oven gas and superheated steam was introduced into the top of the oven to minimize cracking of the pitch gas and vapors. The over-all oven time for one charge of pitch was about 22 hours. The coke was pushed from the oven into a quencher car and was discharged from the latter onto a loading wharf. To avoid the use of an excessive amount of water in the quenching operation, a cover on the quencher car was lowered into position after the necessary amount of water had been applied to the coke. Quenching was then effected principally by the steam formed by evaporation of the quenching water.

The vapors from the ovens passed through electrical precipitators, indirect coolers, and direct coolers to remove the pitch distillate. The noncondensable gas was pumped to gas holders. The pitch distillate was shipped to Hamburg for the manufacture of a road binder (Ebano Spezialbindemittel), and the gas was used for heating and purging the ovens, for prewarming the oven doors, heating the charging tanks, and generation of steam for the pitch-coke plant.

In 1943, a second battery of 20 ovens was installed exactly like and in line with the first battery. Operation of the new battery began in November of that year. The primary purpose of this second battery was not to double the capacity of the plant but to insure uninterrupted operation at the full capacity of one battery if part or all of the capacity of either battery should be lost for any reason.

During the period between December 1939 and January 1, 1945, this plant produced 342,000 tons of pitch coke. The pitch-coke plant suffered considerable damage by bombing. One block of five ovens received a direct hit, and during the last month of the war the equipment was heavily damaged by artillery.

Ruhröl G.m.b.H. Bottrop-Welheim. - So great was Germany's need for pure carbon for electrode manufacture that other sources, in addition to the two pitch coke plants described above, were sought. At the Bottrop-Welheim plant of Ruhröl G.m.b.H., a process was employed that produced electrode coke with an unusually low ash content (0.10 to 0.15 percent). For this reason, it commanded a somewhat higher price than the cokes from other sources.

The process discussed at length by Lowry and Rose in C.I.O.S. Report, Item No. 30, File No. XXXI-27, consisted in (1) digesting bituminous coal at about 400°C. under 100 to 150 atmospheres pressure in middle oil from the liquid-phase hydrogenation of a 70/30 mixture of briquette pitch and coal-tar oil; (2) filtering the digestion product through special ceramic filters at about 150°C. and under 4 to 6 atmospheres pressure to remove ash and carbonaceous material not dissolved by the extracting medium; (3) distillation of the filtrate to obtain the coal extract (softening point 210°-220°C.) and to obtain a distillate for gas-phase hydrogenation and re-use as extracting medium; (4) cooling of the coal extract on an endless steel conveyor; and (5) carbonization of the solid coal extract in byproduct coke ovens to obtain low-ash coke of suitable quality for carbon electrode manufacture.

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Zeche Carl Alexander, Baesweiler, near Alsdorf. - Another source of material for the production of carbon electrodes was the Carl Alexander plant at Baesweiler, where specially selected bituminous coking coal of low ash content, after screening and preflotation, was subjected to four successive flotations, using beechwood tar oil as the flotation reagent. The final froth from the flotation process went to carbon-lined, concrete tanks, where it was extracted with a solution containing 100 parts water, 2 parts hydrochloric acid, and 1 part hydrofluoric acid. The ratio of acid solution to coal in the extraction process was 2:1. The ash content of the coal was reduced in this manner to 0.5 percent. After extracted coal had been recovered from the acid solution by filtration, washed with water, again filtered, and dried, it was carbonized in byproduct coke ovens without the admixture of pitch or other binding materials.

The amount of clean coal made at this plant for electrode coke production was about 6,000 tons per month. The process has been described in greater detail in FIAT Final Report No. 634, page 42, by Fraser and Driessen.

Mannesmannrohrenwerke, Zeche Königen Elizabeth, Essen. - Another coal-cleaning process for making raw material suitable for carbon electrode coke manufacture was under development at the Königin Elisabeth plant of Mannesmannrohrenwerke A.G. near Essen. It also has been described by Fraser and Driessen in FIAT Final Report 634, page 48. This process consisted in picking, crushing, and dedusting bituminous coal from the Finefrau seam, followed by laminar separation (7-1/2 tons per hour) in a Vogel-Laminarström washer using a suspension of magnetite in water as the separating medium. The cleanest coal from this operation (5 to 6 tons per hour) contained about 1.2 percent ash. After rinsing on a horizontal Schieferstein resonance screen and recovery of the suspension medium by magnetic separation, the coal was milled in a ball mill in closed circuit with a rake classifier and the fines from this operation went to flotation cells, the flotation froth was broken and dewatered, and the "purest coal" was shipped to coke plants, where it was mixed with 30 percent hard pitch (150°C.) and carbonized in byproduct coke ovens. The ash content of the "purest coal" was about 0.5 to 0.7 percent.

Another system of cleaning coal from the Finefrau seam for use in carbon electrode manufacture was under development at Königin Elisabeth. It involved the use of a Büttner dryer and electrostatic separation. The equipment was not ready for trial before the war ended.

Special Pitch Compounds

Interest in special pitch compounds of the type called Plastix increased somewhat during the war. This was due in part to the almost complete lack of asphalt (bitumen) for those purposes for which it had previously been employed, such as paving, roofing, flooring, waterproofing, insulating, and pipe-line protection.

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Plastix were made by fluxing coal-tar pitches having softening points above 100°C. (K.S.) with high-boiling distillates or combinations of distillates, such as filtered anthracene oil, oils recovered in distilling briquette pitch to hard pitch, and oil of the type used for the recovery of light oils at coke plants (wash oil). These hard pitch-heavy oil mixtures have better temperature-viscosity relationships than straight distilled coal-tar pitches and are therefore better suited to uses requiring resistance to flow at elevated atmospheric temperatures or less brittleness at low temperatures. This characteristic is indicated, in German practice, by the spread between the softening point (Kramer-Sarnow) and the breaking point as determined by the Fraas test. According to one manufacturer of Plastix, the spread between softening point and breaking point should be at least 40°C.

Rütgerswerke A.G. - Rauxel. - The method used by Rütgerswerke A.G. for producing Plastix has been mentioned above under hard-pitch production. Briefly, it consists in charging 70 to 80 tons of soft pitch into the special Plastix-making equipment, heating the pitch to 290° to 300°C, then blowing with approximately 400 to 600 cu.m. of air per hour, and a small amount of steam with continued heating until the desired softening point of 180°-200°C. is reached. At that time the temperature of the pitch should be at least 350°C. The time required for the blowing operation is about 10 to 14 hours. The high softening point pitch is fluxed to the desired final softening point, generally between 38° and 60°C., with filtered anthracene oil or, preferably, with a mixture of oils containing about 70 percent of anthracene oil having a distilling range of 350° to 450°C. and free from crystalline solids, and 30 percent of wash oil with a distilling range of 250° to 320°C., also free from crystalline material. For certain grades of Plastix, such a mixture of oils and the high softening-point pitch are mixed in approximately equal quantities.

Pluto-Wilhelm, Wanne-Eickel. - To a considerable extent, Chemical Works Pluto-Wilhelm, of the Gelsenkirchen Mining Co., specializes in making special pitch compounds of the type called Plastix. Coal tar is distilled, with vacuum, to softening points above 100°C. The hard pitch is fluxed with a mixture of heavy anthracene oil distilling about 270°C., and the fraction of oil recovered from tar on distilling briquette pitch to hard pitch. Various proportions of pitch and oil mixtures are used for different purposes. A base containing 56 percent hard pitch and 44 percent of the oil mixture is used in making filled compounds in which the filler is stone dust, talc, asbestos fines, fly ash, or slag wool. The same base also is fluxed with volatile solvents for cold application purposes.

Gesellschaft für Teerverwertung, Rauxel. - At Rauxel, Gesellschaft für Teererzeugnisse made Plastix by fluxing hard pitch with hard-pitch distillate in a 36 cubic-meter, steam-heated tank using air for agitation.

Some other German tar plants in addition to those mentioned above make pitch compounds of the Plastix type for roofing, waterproofing, pipe coating, joint filling, and other purposes for which pitches having improved thermal characteristics are desired.

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Road Tars

The construction of new roads in Germany was abandoned almost completely during the war. Such road tars as were made principally for maintenance purposes were mostly of the types developed and standardized in prewar years. However, a special type of road tar was made by one company in substantial quantities as the result of circumstances brought about by war conditions.

Ebano Asphalt Werke Aktiengesellschaft. - Ebano Asphalt-Werke Aktiengesellschaft at Hamburg-Harburg, when it could no longer supply asphalt for paving purposes, was asked by the German Government to make a compound suitable for such use from stocks of briquette pitch, hard-pitch distillate, and pitch-coke distillate that were available at the time. The hard-pitch distillate was the oil produced in distilling briquette pitch to hard pitch, and the pitch-coke distillate came from the Rauxel pitch-coke plant.

A mixture, called "Ebano Spezialbindemittel," was made, which at first contained no asphalt. Later, asphalt from oil distillation in Roumania was added when supplies of that material became available. Following are the percentage compositions of typical mixtures prepared in 1941, 42, 43, and 44. The quantities made are also given in the table.

	1941	1942	1943	1944
Hard-pitch distillate	35.1	36.2	10.3	26.5
Pitch-coke distillate	32.2	39.3	29.4	11.9
Soft pitch, 40°-50°	16.6	4.5	16.1	17.5
Briquette pitch, 65°-70°	7.9	9.5	14.2	17.5
Low-temperature tar from Silesia	4.5	7.4	2.6	-
Tar acid free tar oil	3.7	3.1	6.6	-
Roumanian asphalt	-	-	18.3	15.5
Crude coal tar	-	-	-	3.1
Slopol	-	-	2.5	-
Anthracene oil	-	-	-	8.0
Tons produced	52,256	59,931	56,080	27,054

The equipment used for making Ebano Spezialbindemittel consisted of two tanks of 1,000 tons capacity each connected through a pump and equipped with air-agitating coils. After one of the tanks had been filled with pitch or a pitch-asphalt mixture and the other with the mixture of oils and tars, the contents of the two tanks were mixed by circulation with the pump and by air agitation.

During 1943 and 1944, an emulsion was made from Spezialbindemittel by mixing 35 percent of it with 50 to 55 percent water and 10 to 15 percent of a mixture containing one-third clay similar to bentonite and two-thirds common clay in horizontal, mechanical mixers. Camouflage paint was made from the emulsion by mixing 40 percent emulsion with 20 to 30 percent mineral pigment and 30 to 40 percent water. The production of Spezialbindemittel ended abruptly in 1944 when the plant of Ebano Asphalt Werke was heavily damaged by air raids. The equipment for making Spezialbindemittel was destroyed completely.

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Carbon Black

The much publicized carbon-black industry of Germany, which produced blacks for the rubber industry during the war, was largely made possible by the fact that large quantities of crystal-free, high-boiling tar oils were needed for fuel purposes and smaller quantities for the fluxing of the special compounds such as Plastix and Spezialbindemittel, which have been discussed above.

The usual practice in German tar plants when distilling coal tar is to collect anthracene oil as a separate fraction or in two separate fractions, light and heavy. On cooling to atmospheric temperature, a mixture of crystalline solids settles out. When the oil is to be used as fuel by the Navy or others, especially in combination with other liquid fuels, such separation of solids is troublesome. The crystalline solids also make the oil less suitable for fluxing pitch compounds and road tars, so most German tar plants crystallize the anthracene oil and filter or centrifuge it to remove the mixture of solids, which they call "anthracene rückstand" or anthracene residue. The equipment used by Gesellschaft für Teerverwertung for this purpose has been described.

Refined anthracene can be obtained from anthracene residue, but the market for refined anthracene in Germany was not large enough to absorb all of the residue. Having no other outlets for it, the tar distillers sold it to the carbon-black plants, which considered it to be the most suitable raw material for their purpose, chiefly on account of its comparatively low price. Anthracene oil could be used interchangeably with anthracene residue at the carbon-black plants but cost 12.0 RM per 100 kilos, as compared with 5.4 RM for anthracene residue. Naphthalene, in addition to costing 9.0 RM for the 76° grade and 11.5 RM per 100 kilos for the hot-pressed grade (78°), required higher pressure steam (20 to 25 atmospheres) than anthracene residue, and also required different combustion conditions. Because of these two requirements, naphthalene could not be used at the largest carbon-black plant, at Dortmund, but it was used at the original and smaller plant at Kalscheuren near Cologne.

The process of making carbon black from coal-tar hydrocarbons was developed before the war by Deutsche Gold und Silber Scheide Anstalt at Frankfurt am Main. Plants were installed at Kalscheuren, near Cologne, at Dortmund-Ruhr, and at Gleiwitz in Silesia. At the Dortmund plant (Deutsche Gasrusswerke G.m.b.H.), maximum production was reached in 1942. The plant was used to the extent of 90 percent of its capacity in that year and produced 14,063 tons of carbon black.

Deutsche Gasrusswerke (G.m.b.H.). - The process, as operated at Dortmund, was as follows: Anthracene residue received from tar-distillation plants in open cars was unloaded into a storage bin, from which it was charged into steam-heated melting kettles by means of a clamshell bucket. The bucket could be moved to any part of the bin and to the feed chute of any melting kettle. The molten anthracene residue was transferred to vertical storage tanks, from which it flowed to vaporizers. Each vaporizing unit

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was equipped with a device for metering and feeding the proper quantity of molten anthracene residue into the vaporizer. Vaporization was effected in the presence of preheated coke-oven gas by steam coils. The mixture of hydrocarbon vapors and coke-oven gas passed through gas heated pipes to the carbon-black machines. In the latter, the vapor-gas mixture was burned with a smoky flame by thirty burner tips located in a row below a steel cylinder through which hot water was circulated. The cylinder was 540 mm. in diameter and 5,000 mm. long. Carbon black deposited on the rotating cylinder (1 r.p.m.) was removed by scrapers and dropped into a screw conveyor, which transported it to other conveyors running to storage bins. The combustion gases leaving the machine passed through ducts to woolen filter bags, which recovered the carbon black that had not deposited on the rotating cylinder. The black from the cylinder was classed as first-quality and that from the filter bags as second-quality.

Lubricating Oils and Greases

Pluto-Wilhelm, Wanne-Eickel. - High-boiling tar oils similar to those used by Chemical Works Pluto-Wilhelm for the making of Plastix were also used by that concern for making lubricating oils and greases. The Pluto-Wilhelm plant has a large and well-equipped department for lubricating-oil and grease manufacture. Before the war, petroleum products were purchased for use in the various formulations. During the war, when petroleum products were less available, tar oils were used in their place in some of the lubricating materials. Medium-heavy anthracene oil 270° to 300°C. and heavy anthracene oil 300° to 360°-400°C. were used directly in greases. For lubricating oils, a mixture of medium-heavy and heavy anthracene oil was redistilled to remove oils distilling below 300°C., and the residue from the distillation was crystallized and vacuum-filtered to remove any remaining portion of the "anthrasen rückstand." Three kinds of tar-grease oil were prepared:

I - 1.2° to 1.6° Engler at 50°C.
II - 1.6° to 2.2° Engler at 50°C.
III - 2.75° to 3.2° Engler at 50°C.

Lubricants were made by dissolving vegetable or animal fats or fatty acids in tar oils or mixtures of tar oils and mineral oils in steam-heated kettles. The solution so prepared was then boiled with milk of lime to form a soap, which was diluted with mineral oils or tar oils, and, if necessary, other raw materials were added to obtain the desired consistency. The lubricant was then run into cooling tanks, where it was cooled by stirring until the proper structure was reached, after which it was filled into containers.

To make emulsifiable boring, cutting, and cooling oils, procedures similar to the above were followed, but tar-oil sulfonates or emulsifiable waxes were added to the other ingredients.

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Puttys were prepared by mixing chalk or other mineral substances with linseed oil or drying tar oils made from benzol refining-still residues. Also, puttys were made with combinations of mineral substances with drying oils and resin pitches or tar pitches. A kneading mill was used for the putty manufacture.

Lubricating oils and greases, made as described above, were used principally in heavy-duty applications such as the lubrication of elevator guide rails in mine shafts, and for such purposes they were considered to be satisfactory.

Rheinpreussen, Moers-Homburg. - Lubricating oils containing naphthalene instead of tar oils were manufactured by Rheinpreussen at Moers-Homburg. The process used at that plant, as described by Atwell and Schroeder in CIOS Report Item 30, File XXIV-9, was as follows: "Fischer Tropsch middle oil, boiling range 250°-350°C., is chlorinated at 80°-100°C. to the extent of 20 to 25 percent chlorine by weight. Five volumes of chlorinated oil are reacted with two volumes of naphthalene at 70°-100°C. in the presence of eight volumes of Fischer-Tropsch benzin and a small amount of $AlCl_3$ as catalyst. The sludge is withdrawn, the oil is neutralized with lime and bleaching earth, filtered, and stripped free of benzine. Subsequent vacuum distillation yields middle oil and turbine oil overhead and cylinder stock as bottoms. The fractions are used as such and are also blended to make motor oils."

According to Atwell and Schroeder "all products were stated to have a high viscosity index and great resistance to oxidation, but tests on captured samples do not support these claims." When interrogated concerning lubricating oils made in this manner with Fischer Tropsch oil and naphthalene, Dr. Martin, director of Ruhrchemie at Sterkrade-Holten, expressed the opinion that they were inferior, from the standpoint of thermal characteristics, to lubricating oils made from Fischer-Tropsch oils alone.

Hydrogenation of Pitch and Tar Oils

In 1943, the German tar plants supplied to hydrogenation plants 215,073 tons of pitch, 171,389 tons of heavy tar oils, 19,520 tons of hard pitch distillate, and 2,897 tons of pitch-coke distillate. Altogether, the quantity of pitch and oils furnished for this purpose was 408,859 tons, or approximately 89,950,000 U.S. gallons.

Ruhröl G.m.b.H. - The tar oil went to various hydrogenation plants, but a large proportion of it and substantially all of the pitch went to the one hydrogenation plant at Bottrop-Welheim operated by Ruhröl G.m.b.H. By the early part of 1943, that plant could hydrogenate 263,000 tons per year of a mixture containing 70 percent briquette pitch and 30 percent tar oils. The products from that quantity of mixture were 120,000 tons of heating oil suitable for use by the navy, 60,000 tons of aviation gasoline, and 18,300 tons of fuel gas (propane and butane). The navy fuel oil was claimed to be completely free of asphalts and, because of its aromatic

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character, to have good asphalt mixing or dissolving capacity when used with inferior fuel oils. The aviation gasoline had an octane rating of 92 and was said to perform well in supercharged motors.

Hydrogenation of the pitch-tar oil mixture at Welheim was carried out in two stages, in each of which the pressure was 700 atmospheres. Liquid-phase hydrogenation was used in the first stage to produce the Navy fuel-oil and gas-phase hydrogenation yielded the aviation gasoline in the second stage. Fuel gas came from both stages of the process. A complete description of the operation at Welheim has been given by C. Cockram in CIOS Report Item 30, File XXX-104.

The operation of the Welheim plant was stopped by an air raid in July, 1944. After 3 months of reconstruction it was ready to start operating again but was shut down completely by five air raids between October 30 and November 30, 1944.

Coal Briquetting

The increased use of pitch for heating, hydrogenation, and carbon-electrode manufacture from 1940 to 1944 caused a corresponding decrease to take place in the use of pitch for the briquetting of coal fines. This change is indicated by the following table, in which the percentage distribution of high-temperature coal-tar pitch in 1943 is contrasted with the percentage distribution in 1940.

Percentage distribution of coal-tar pitch by uses

Uses	1940	1943
Briquettes	47.8	34.4
Carbon electrodes	15.6	20.9
Hydrogenation	5.9	17.2
Roofing felt	11.4	10.3
Road building	7.7	2.8
Heating and carburetion	3.8	5.3
Other uses	7.8	9.1

Ruhröl G.m.b.H. - No important changes were made in methods for producing or using briquette pitch, with one exception, which resulted from the hydrogenation of pitch and tar oils at the Welheim plant of Ruhröl A.G. Sludge amounting to about 15 percent of the pitch-tar oil feed was withdrawn from the liquid-phase portion of the system and was transferred to the tar-distilling plant of the Mathias-Stinnes organization, where it was added to coke-oven tar in the proportion of 140 tons of sludge to 100 tons of tar. The mixture was distilled to briquette pitch. The yield of briquette pitch from the sludge component of the mixture was about 90 percent.

Investigational work was carried out, especially by the Mathias-Stinnes organization, to determine to what extent, if at all, the quality of the briquette pitch was altered by the admixture of hydrogenation sludge to the tar. Tests showed that the briquette pitch, so made, was slightly inferior

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to normal briquette pitch but complied with standard specifications and was acceptable to the briquetting industry.

The amount of hydrogenation sludge contained in briquette pitch in 1943 was approximately 17,000 tons, or about 4 percent of the total briquette pitch used in that year. About half as much hydrogenation sludge was used in this manner in 1942 and substantially none in 1941. At that time efforts were still being made, without appreciable success, to operate the Welheim hydrogenation plant on coal extract produced by the extraction of coal with a mixture of tetraline and cresylic acid.

Roofing and Waterproofing Products

No important changes were made during the war in methods for producing tar-saturated and coated felt for roofing and water-proofing purposes except possibly for an increased use of coatings made by procedures like those employed for making Plastix, i.e., by the fluxing of pitches with heavy oils followed by the addition of mineral filters.

In the German roofing-felt plants, the dry felt is first impregnated with a coal-tar saturant and is then coated with a soft pitch or Plastix-type compound, to which sand is applied as an antistick and weather-protective coating. Felts coated with the Plastix type of coating have greater flexibility at low temperature than those coated with ordinary coal-tar pitches.

New or Increased Uses of Chemicals from High-Temperature Coal Tar

It appears that little or no research was carried on after 1938 in laboratories associated either directly or indirectly with the German high-temperature coal-tar-distilling industry with a view to making new or improved products from high-temperature coal tar. Procedures for the recovery of the various chemicals and products furnished by the tar distillers after 1938 had, for the most part, been worked out in previous years. Also, little or no research on high-temperature coal tar or products derived therefrom appears to have been carried on by research institutions that might ordinarily be expected to include such work in their research programs.

Undoubtedly, some research involving the use of chemicals derived from high-temperature coal tar was carried out in the laboratories of major industrial concerns, but, with the exception of pyrene, carbazole, symmetrical xylenol, and diphenylene oxide, such research does not appear to have reached the stage where commercial quantities of unusual coal tar chemicals were required.

On the other hand, large quantities of the usual coal-tar chemicals were produced at the tar plants.

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Naphthalene. - The total quantity of naphthalene sold in 1943 was about 74,000 tons (metric). It was distributed approximately as follows: Chemical uses by I.G. Farbenindustrie, 30,000 tons; hydrogenation to tetralin and decalin by Deutsche Hydrierung A.G., 13,000 tons; chemical uses by others, 4,073 tons; carbon black, 15,600 tons, and lamp black, 2,400 tons; insecticides 4,600 tons; preservatives, 2,300 tons; lubricants (Rheinpreussen), 1,700 tons; and miscellaneous, 400 tons, including plant protection materials, insulating brick manufacture, and Diesel fuel.

The chemical uses by I.G. Farbenindustrie included the manufacture of beta naphthol and other dye intermediates, phthalic anhydride, plasticizers, tanning agents, preservatives, wetting agents and an emulsifier for Buna rubber. Of these uses, those that were comparatively new in Germany were phthalic anhydride manufacture and the production of the emulsifier for Buna (Nekal B.x or Emulgator 1000).

The manufacture of phthalic anhydride by I.G. Farbenindustrie at Ludwigshafen and Schkopau and the construction of a plant for its production at Uerdingen has been discussed at length in the following CIOS reports:

Ludwigshafen, Item No. 22, File No. XXVII - 85
Schkopau, Item No. 22, File No. XXVIII - 29
Uerdingen, Item No. 22, File No. XXVII - 80

Phthalic anhydride was made in the same manner at Ludwigshafen and Schkopau. The capacities of these two plants were approximately 600-700 tons and 500-600 tons of phthalic anhydride per month, respectively. The capacity of the Uerdingen plant was expected to be about 300-400 tons per month. Its equipment was approximately the same as that used in the other two plants. The process at all plants consisted in oxidizing naphthalene with air in the presence of a vanadium pentoxide catalyst containing potassium sulfate. Salt-cooled converters were used.

Nekal B.X., mentioned above, was first made in Ludwigshafen in 1917, but new plants for its production were installed during the war at Schkopau, Hüls, and Auschwitz. At Schkopau it was called Emulgator 1000. The capacity of the Schkopau plant alone was about 300 tons per month. This chemical is the sodium salt of dibutyl naphthalene sulfonic acid. It was said to be the best emulsifier in Germany for use in the manufacture of Buna rubber.

The use of 13,000 tons of naphthalene in 1943 by Dehydag (Deutsche Hydrierung A.G.) for the production of tetralin and decalin was mentioned above. Those chemicals, in turn, were used by the Wehrmacht (for the Navy, Army, and Air Forces), by the railroads for heating and lighting, for the extraction of naphthalene from gas, and in lacquer manufacture.

Phenol and cresol. - The recovery of phenol and cresols from high-temperature tar and from the ammonia liquors from coke plants amounted to about 13,000 tons in 1943. They were used for making resins, dyestuffs, plasticizers, pharmaceuticals, and for other well-known purposes.

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One extensive use for phenol consisted in the manufacture of the so-called Igamids, involving, as intermediate steps, the hydrogenation of phenol to cyclohexanol, dehydrogenation of cyclohexanol to cyclohexanone, conversion of cyclohexanone into the oxime with a solution of hydroxylamine sulfate, reaction of the oxime with sulfuric acid followed by neutralization with ammonia to obtain caprolactam and polymerization of the latter to the Igamids. The various steps mentioned above have been described in greater detail in CIOS Report Item 22, File XXVI-53, by Hasche and Boundy.

A minor use for phenol was the manufacture of Koresin, although at Ludwigshafen the production of this chemical amounted to 120 to 140 tons per month. The process, described in CIOS Report Item 22, File XXIX-62, consisted in reacting isobutylene with phenol, using $AlCl_3$ as catalyst, to obtain para tertiary butyl phenol. The latter was then reacted with acetylene under pressure at 230°C., using zinc naphthenate as catalyst. The product (Koresin) was used as a tackifier for Buna.

Pyridine bases. - Uses for pyridine bases in Germany were not numerous. The total quantity distributed in 1943 was about 950 tons, of which about 190 tons were used in the dying of fabric and in wetting agents, 350 tons were used by I.G. Farbenindustrie in part for refining anthracene and carbazole, 260 tons were employed for denaturing alcohol, and 130 tons for other purposes.

Some investigational work was done by I.G. Farbenindustrie at Leverkusen on the production of pyridine from higher boiling bases. Although the yield was only about 50 percent of theoretical, it was planned to build a larger pilot plant. The war caused this plan to be abandoned. Vapors of the mixed bases with hydrogen and a small amount of H_2S were passed through a nickel-chrome steel tube at about 860°C.

Pyrene. - In 1943 Rütgerswerke A.G. furnished about 120 tons of pyrene to I.G. Farbenindustrie for the manufacture of dyestuffs. The pyrene was produced by fractionation and crystallization from high-boiling coal-tar oil.

Documents indicate that I.G. Farbenindustrie was interested in the use of pyrene for dyestuff manufacture before the war and carried on some research with a view to producing it synthetically. Apparently this development was not successful or was not carried to completion, but some interest in the use of pyrene from tar continued throughout the war, as indicated by the purchases mentioned above.

Carbazole. - In 1943, Rütgerswerke A.G. also furnished 127 tons of carbazole to I.G. Farbenindustrie. It was recovered from a crude carbazole fraction by crystallizations from solvents followed by sublimation. The carbazole was used for the manufacture of 1,3,6,8 tetranitro carbazole. The latter was used in the production of an insecticide, which, according to CIOS Report Item 24, File XXVII-4, was used in vineyards in place of arsenical insecticides against the vine moth.

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Nirosan contained 25 percent of 1,3,6,8 tetranitro carbazole, 10 percent of sulfite pulp, 0.25 percent wetting agent (Igepal), and kaolin or chalk. Carbazole for this purpose and for the manufacture of dyes was of considerable interest to I.G. Farbenindustrie. According to CIOS Report Item 22, File XXVI-11, research was conducted at Höchst on methods for its synthesis. The report states that "the synthesis of carbazole did not look promising, and no further work was contemplated."

Insecticides from symmetrical xylenol. - Symmetrical xylenol, chlorinated by Rütgerswerke A.G. at Niederau and by Dr. F. Raschig G.m.b.H. at Ludwigshafen, was supplied to manufacturers of disinfectants. 150 tons of symmetrical xylenol was produced by Rütgerswerke A.G. at Rauxel in 1943 for chlorination at Niederau and subsequent shipment to insecticide producers in Hamburg. One disinfectant of this type, made by Schulke and Mayr, Hamburg, was called "Grotan."

Diphenylene oxide. - Rütgerswerke sold 144 tons of diphenylene oxide through brokers in 1943, but the manner in which this product was used is not definitely known. It has been stated that it was used as a base for scents and for dyestuffs.

Acenaphthene and chrysene. - 263 tons of acenaphthene were supplied by Rütgerswerke to CIBA for dye manufacture, and 8 tons of chrysene went to I.G. Farbenindustrie for the same purpose.

Methyl naphthalenes. - Some alpha methyl naphthalene was used as cetane reference fuel, and 5 to 10 tons of beta methyl naphthene went into the manufacture of plant growth-promotion chemicals.

Miscellaneous chemicals. - In addition to the above-mentioned chemicals, many others were supplied, principally by Gesellschaft für Teerverwertung, but the quantities were small and were used mainly for research purposes.

PART II. - COMPANY REPORTS

Reports of visits to organizations interested directly, or indirectly in the processing of high-temperature coal tar and use of products derived therefrom. Producers and users of high-temperature coal-tar products, selling organizations, engineering companies, equipment manufacturers, and research organizations were visited.

Gesellschaft für Teerverwertung Duisburg-Meiderich

The tar-distilling plant of Gesellschaft für Teerverwertung at Duisburg-Meiderich was visited on September 18, 1945. The persons interviewed were Dir. E. Moehrle, Dr. Seidler, Dr. Dorfler, and Paul Von den Hövel. On other visits made to the same plant at later dates, Dr. W. Fischer and Dr. O. Kruber were interviewed in addition to the above mentioned persons.

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This plant was heavily damaged during the latter part of the war. It was about 40 percent destroyed. Two separate tar-distilling units, anthracene buildings, two naphthalene buildings, the benzol plant, and storage tanks suffered the most damage.

Light oils, tar-acid oils, coumarone-bearing oils and residues, and other crude materials are received from other plants of the same organization and from other companies. They are processed at Meiderich to make refined products. The scope of its operations may be judged by the following list of products made in 1938 and 1944 by Gesellschaft für Teerverwertung.

Products (tons per month)	1938	1944
Briquette pitch	11,303	9,134
Prepared tars and road tars	2,776	3,256
Fuel oil	2,264	4,403
Motor fuel oil	300	18
Creosote oil	2,521	-
Wash oil	753	890
Hydrogenation oil	1,740	1,838
Benzene	15	12
Toluene	158	115
Xylene	15	38
Motor benzole	267	184
90 benzole	11	53
Solvent naphtha	129	99
Heavy naphtha	192	108
Coumarone resins	113	98
Phenol	212	359
Cresols	441	535
Xylenols	44	19
Anthracene residue	862	478
Naphthalene, pure	304	319
Naphthalene, hot pressed	303	369
Naphthalene, washed	827	727
Pyridine bases	26	24
Ammonium sulfate	39	23
Roofing felt (in rolls)	7,360	3,680
Acenaphthene	4	-
Special road tar	398	162
Special tar products	167	291
Refrigerated tar oils	265	261
Flaming oil	6	186

No pitch harder than briquette pitch was made at this plant, and no special pitch compounds of the type called Plastix were made.

Changes in processes or equipment made in the last 10 years were as follows: A new continuous tar still, improved anthracene-cooling equipment, continuous crude phenol dehydration, new tar-acid-refining stills, and new oil-redistillation stills.

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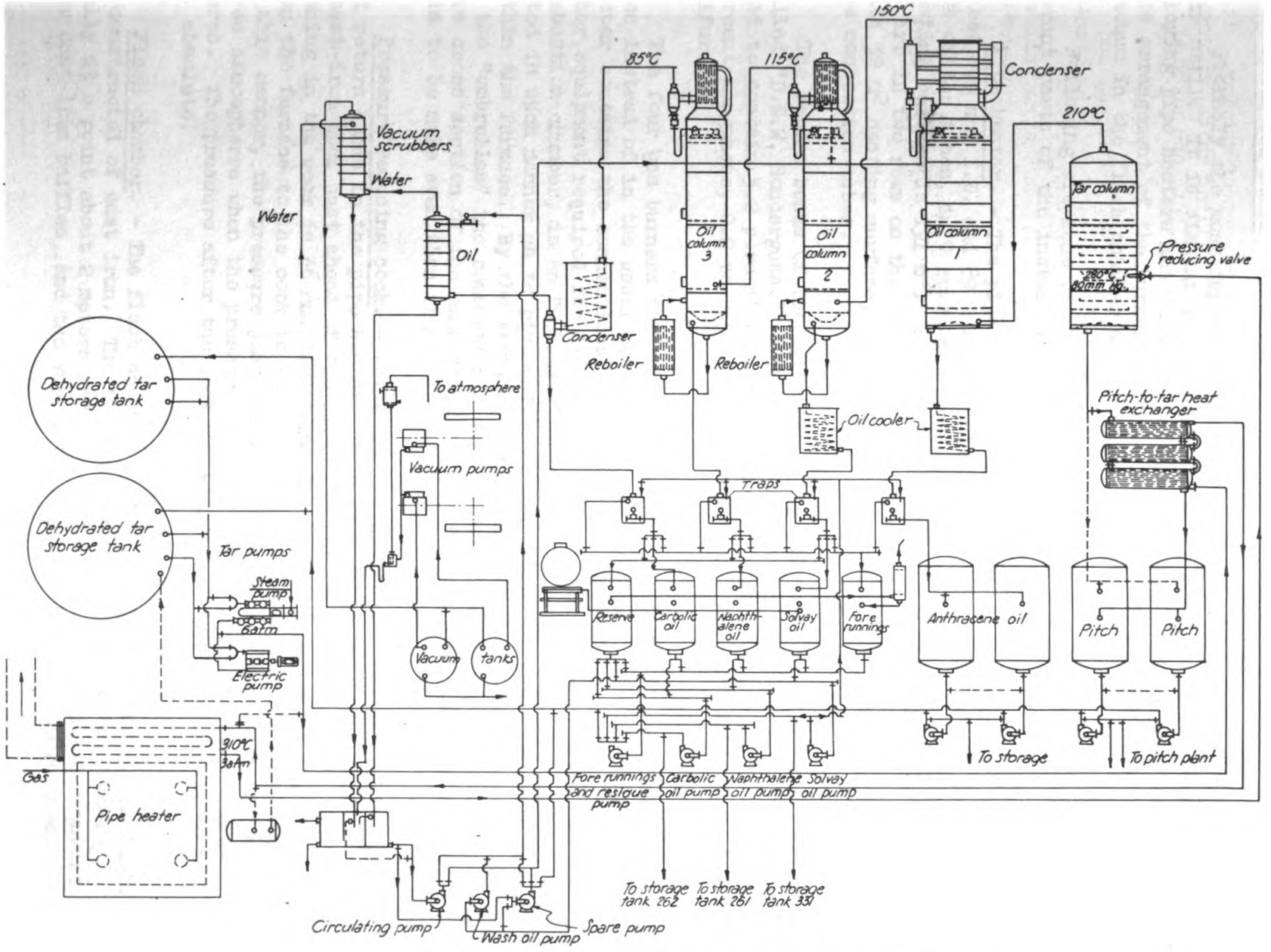

Flow diagram for 6 f.t. continuous tar still, Druisburg-Meiderich

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Continuous Tar Still

Probably the most important innovation at this plant is the continuous tar still. It is similar in principle to other continuous tar stills employing pipe heaters and multiple columns, but differs from them chiefly in the arrangement of the burners in the pipe heater, and the use of high vacuum in the flash chamber and fractionating columns.

Following are some of the more important details concerning the different parts of the installation:

Pipe heater. - The pipe heater of the G.f.T. still contains 190 steel tubes, 79 mm. I.D. and 89 mm. O.D. The total heating surface is 349 m^3. 104 of the tubes, in 9 rows, are contained in the convection bank. Their heating surface is 191 m^2. 66 of the tubes with a heating surface of 121 m^2 are in two rows on the ceiling of the furnace, and 20 tubes, in two rows with 32 m^2 heating surface, are on the upper part of the side wall opposite the convection tubes.

The return bends of the pipe heater are made of a special cast iron called "D.E.W. Sonderguss." Formerly it was called "Emmelguss." It is said to contain 3.2 percent carbon, 1.3 to 1.5 percent silicon, 0.5 to 0.6 percent manganese, 0.2 to 0.5 percent phosphorous, and 0.05 to 0.08 percent sulfur.

The four gas burners for the pipe heater are in the floor of the furnace instead of in the usual position at one side. A room below the pipe heater contains the connections to the burners, the valves, meters, and other equipment required for its control. Over each burner, inside the combustion chamber, is an "umbrella" made of fire brick with openings located in such manner as to give uniform distribution of the combustion gases within the furnace. By the vertical arrangement of the burners and the use of the "umbrellas" the combustion gases are spread uniformly over the entire cross section of the combustion chamber and the heat distribution is said to be more even than that of other pipe-heater installations.

Pressure reducing cock. - While the tar is passing through the tubes and return bends in the pipe heater, it is kept under pressure by means of a cast-iron cock just ahead of the flash chamber or "Teerkolonne." The opening in the cock is 36 mm. diameter. The inside diameter of the pipe from the furnace to the cock is 79 mm. By constricting the flow of the tar in this manner, the pressure just ahead of the cock is maintained at two to three atmospheres when the pressure at the tar feed pump is 6 to 7 atmospheres. The pressure after the cock and within the flash chamber is 80 mm. Hg. absolute.

Flash chamber. - The flash chamber, 2.8 m. in diameter and 8.0 m. high, is constructed of cast iron. The tar from the pipe still enters tangentially at a point about 2 meters from the bottom. The pitch flows downward over cast-iron baffles, and the vapors pass upward through a compartment

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containing cast-iron baffles, which serve as entrainment catchers. In the upper compartment of the flash chamber are steel Raschig rings, which serve a dual purpose. They prevent the carry-over of entrained pitch in the vapors entering the first fractionating column and, by reacting with the corrosive elements in the vapors released in the flash chamber, they prevent corrosion of the columns and condensers through which the vapors pass after leaving the flash chamber. This permits all of the apparatus after the flash chamber to be made of ordinary steel. After about 8 months of operation, the Raschig rings are removed from the flash chamber. Those that are severely corroded are replaced, and the rest are cleaned and returned to the flash chamber.

Fractionating columns. - The three steel fractionating columns, each 10 meters high, have the following diameters: No. 1, 2.8 m.; No. 2, 2.0 m., No. 3, 1.6 m. Column 1 is packed with 50 mm. steel Raschig rings. Columns 2 and 3 are packed with 35 mm. Raschig rings.

In each column the packing is contained in two compartments, which can be emptied separately. The dephlegmator on column 1 is of a cellular type with 22 m^2 surface area. Those on columns 2 and 3 have vertical tubes with 20 m^2 surface area each. Reflux is returned by gravity from each dephlegmator onto the packing within the column by means of a circular trough with vertical slots on the inner and outer side walls. There are 24 slots in the inner wall of the trough and 48 slots on the outer wall. The slots are 125 mm. deep and only a few mm. wide. Welded to the bottom of the trough is a curved ring, which forms two sloping surfaces over which the reflux liquid from the slots flows onto the surface of the packing. The liquid drops onto the packing in two substantially continuous rings. The diameter of the inner ring is 1,120 mm. and of the outer ring 2,230 mm. in column 1. The inside diameter of this column is 2,825 mm.

Vacuum system. - The vapors leaving column 3 pass through a water-cooled coil condenser with 43.7 m^2 surface area and then through two bubble-tray columns each 1 m. diameter by 3 m. high. A part of the wash-oil fraction is used as a scrubbing medium in the first column and water in the second. After leaving the second scrubber, the noncondensable gases pass to either of two vacuum pumps of 2,850 m^3 capacity per hour each.

Reheaters. - Figure 1 shows columns 2 and 3 equipped with heaters that were intended to be used as reboilers to strip low-boiling oils from the heavier oils leaving the bottoms of the columns. However, it was found to be unnecessary to supply any heat other than that furnished to the tar in the pipe heater.

Oil coolers. - The oils from the bottoms of columns 1 and 2 pass through water-cooled coils having 13 m^2 cooling surface each before entering the receiving tanks.

Receivers. - All of the condensates flow by means of barometric legs to seal pots, from which they discharge by gravity into five steam-heated

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receiving tanks of 8 m^3 capacity and two of 25 m^3 capacity. The former are used for forerunnings, carbolic oil, naphthalene oil, wash oil, and reserve and the latter for anthracene oil.

Pitch from the flash chamber flows by gravity through a pitch-to-tar heat exchanger to either of two receivers of 25 m^3 capacity each.

Pumps. - The plant is equipped with the following pumps:

1 electric 3-piston pump, 12 m^3/hr., 15 atm. (tar feed).
1 steam, single-piston pump, 10-15 m^3/hr., 15 atm. (tar feed).
6 circulating pumps, rotary, 2 m^3/hr., 2 atm.
2 pitch pumps, electric, special rotary, 2 m^3/hr., 2 atm.

Temperatures, °C. - Feed 100°; pipe still outlet, 320°; flash chamber 280°; overheads of columns 150° - 115° - 95°; pitch 260°.

Reflux ratio for each column. - 1.0-2.0.

Consumption per ton of tar. - Gas, 30 m^3; steam 30 kg; electricity 4 kw.-hr., water 1.5 m^3.

Products. - Pitch 68°C. K.S., 55 percent (of dehydrated tar); anthracene oil, 300°-400°C., 23 percent; wash oil, 240°-285°C., 9 percent (free of crystalline solids); naphthalene oil, 220°-350°C., 6 percent (free of phenol); carbolic oil, 190°-218°C., 6 percent.

Personnel required for operation. - Two men per shift.

Operating time between cleaning periods. - Pipe still, 1 year; columns, flash chamber, 6-8 months; 1,2, and 3, 2 to 3 years; condensers, coolers, etc., 2 to 3 years.

Claimed advantages. - The following claims are made for this installation by Gesellschaft für Teerverwertung (stetige Destillation des Nielstickstoffgemisches Steinkohlenteer, W. Fischer, Zeitschrift V.D.I. - Beiheft "Verfahrentechnik" Nr. 1, 1944, pp. 13 to 29).

1. A heat consumption below all present known values.
2. An increased yield of oil (about 3 to 5 percent).
3. Control of all corrosion influences.
4. Avoidance of coke formation in the heating tubes.

According to the owners, this still cannot be used to make hard pitches (above 100°C. softening point). If this is desired, the briquette pitch made in the continuous still will be transferred to other equipment for further distillation.

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Anthracene Residue Recovery

An improved system of removing the so-called "anthrazen rückstand" or anthracene residue is used at this plant. Twenty water-cooled vertical crystallizers are mounted above five vertical basket centrifuges arranged for mechanical unloading. Each crystallizer is 4,630 mm. high by 2,000 mm. diameter. An outer shell 2,160 mm. in diameter, open at the top, comprises a water jacket. The agitator, driven from above by a 3.7 kw. motor, moves the oil away from the side walls and conical bottom. It is provided with a screw on the lower end of its shaft, which assists in the discharge of the crystalline slurry through the 150 mm. opening in the bottom of the crystallizer.

The five centrifuges, made by C.G. Haubold A.G. at Chemnitz, have phosphor-bronze baskets 2 m. in diameter. Their operating speed is 600 r.p.m. They are equipped with manually controlled mechanical cutters for unloading, and discharge their contents onto conveyors, which transfer the anthracene residue directly into open cars.

The quantity of anthracene oil charged into each crystallizer is 12 cubic meters. The temperature of the oil at time of charging is 80°C., and when ready for centrifuging it is 20°-29°C. The cooling of one charge requires from 1 to 1-1/2 days. After it has cooled sufficiently, the crystalline slurry is fed by gravity from a crystallizer to one of the centrifuges, and the cake is whizzed for 5 to 15 minutes, depending on the quality of the anthracene oil charged to the crystallizer. Normally, the capacity of each centrifuge is about 80 tons of slurry per 24 hours. Anthracene residue produced in this manner contains only 2 to 3 percent of anthracene oil. The anthracene content is 20 to 25 percent. The centrifuged oil remains crystal-free at 0°C.

Crude Phenol Dehydration

The comparatively new equipment at Meiderich for the dehydration of crude phenol consists of two vertical, gas-fired, 10 cubic meter stills equipped with columns 1 x 5 meters packed with iron Raschig rings. The capacity of each still is about 20 tons of crude phenol per day (15 to 20 percent water). The temperature of operation is 160°C., and the gas consumption is 120 to 140 tons per hour, or about 60 cubic meters per ton of crude phenol.

Tar Acid-Refining Stills

The tar acid-refining stills most recently installed at this plant are 50-ton vertical stills fired by gas from below and equipped with columns 1.5 by 10.5 meters, packed with 25 by 25 mm. Raschig rings.

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Oil-Redistilling Stills

Between 1935 and 1940, four gas-fired, 17-ton, vertical stills with packed columns 1 meter in diameter by 5 meters high were installed for the redistillation of oil. In 1940, four additional stills of 50 tons capacity with 1.5 by 10 m. packed columns were installed. The packing rings are iron and 35 by 35 mm.

Special Chemicals

For several years this plant has produced small quantities of special chemicals in laboratory and pilot-plant equipment for sale to universities, research laboratories, etc. This was continued to some extent during the war. In the following table are given the approximate yearly quantities of such chemicals sold by Gesellschaft für Teerverwertung.

Approximate Yearly Production of Special Chemicals Made by Gesellschaft für Teerverwertung, Duisburg-Meiderich.

Acenaphthene, pure	100 kg.
Acenaphthene, tech.	150 tons.
Acridine, pure	50-100 kg.
Anthracene, pure	50 kg.
Quinaldine, pure	10 kg.
Quinoline, pure	500 kg.
Quinoline, tech.	60-80 tons
Chrysene, pure	20 kg.
Collidine (2,4,6), pure	25 kg.
Dicyclopendadiene, pure	200 kg.
Dicyclopentadiene, tech.	5-10 tons
Dimethyl naphthalene, pure, 1,6; 2,6; 2,7; 2,3; each	20-40 kg.
2,4 dimethyl pyridine, pure	a few kg.
2,6 dimethyl pyridine, pure	a few kg.
0,0' dioxy diphenyl, pure	abt. 20 kg.
0,0' dioxy diphenyl, tech.	200 kg.
Diphenyl, pure	100-150 kg.
Diphenyl, tech.	500-600 kg.
Diphenylene oxide, pure	100 kg.
Diphenylene oxide, tech.	200 kg.
Fluoranthene, pure	a few kg.
Fluorene, pure	50-100 kg.
Indene, pure	50 kg.
Indole, pure	220 kg.
Isoquinoline, pure	a few kg.
Lepidine, pure	5 kg.
Methyl indole, (2), (3), and (7) each.	10 kg.
1, methyl naphthalene, pure	200 kg.
1, methyl naphthalene, tech.	10-50 tons
2, methyl naphthalene, pure	500-1,000 kg.
2, methyl naphthalene, tech.	5-10 tons

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Ortho oxydiphenyl, pure	10 kg.
Phenanthrene, pure	50-100 kg.
Phenanthrene, tech.	50-100 kg.
Alpha picoline, pure	abt. 5 tons
Pseudocumol	50-60 tons
Pyrene, pure	20 kg.
Xylenols:	
1,2,4, pure	50 kg.
1,2,4, tech.	500 kg.
1,3,4, pure	5-10 kg.
1,3,4, tech.	1,500 kg.
1,3,5, pure	5-10 kg.
1,3,5, tech.	60 tons.
1,4,5, pure	5-10 kg.
Meta xylene, pure	50 kg.
Ortho xylene, pure	50 kg.

Gesellschaft für Teerverwertung Castrop-Rauxel

The plant of Gesellschaft für Teerverwertung at Castrop-Rauxel was visited on September 17 and on two occasions thereafter. The persons interviewed were Dr. E. Moehrle, managing director of Gesellschaft für Teerverwertung, and Dr. Klemm, director of the Rauxel plant.

This plant was about 50 percent destroyed by bombing and artillery. When visited, it was distilling only about 200 tons of tar per day. (Previously, when operating at full capacity, it had distilled about 1,500 tons per day.) The chief needs of the plant to run at higher capacity were more fuel gas and more crude tar. The stock of crude tar on hand at all of the coke plants in the British zone amounted to about 50,000 tons, and current production was about 350 tons per day.

The stocks of products on hand were as follows (approximately): Oil in process, 3,500 tons; briquette pitch, 39,000 tons; road tar, 60 tons; creosote oil, 600 tons; hydrogenation oil, 500 tons; anthracene residue, 35 tons; hot pressed naphthalene, 40 tons; pitch coke (contaminated), 300 tons.

The accompanying flow diagram shows the methods of operation and equipment normally employed at this plant. The average monthly productions of the plant in 1938 and 1944 are given in the following table:

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Average monthly production, metric tons

Product	1938	1944
Briquette pitch	7,546	7,437
Hard pitch (150°C.) for coking	4,427	9,023
Prepared tars and road tars	3,697	2,179
Fuel oil	7,194	8,526
Motor fuel oil	62	48
Hydrogenation oil	703	1,353
Creosote oil	1,230	1,142
Wash oil	970	1,195
Anthracene, 40 percent	212	135
Anthracene residue	588	870
Acenaphthene	5	-
Carbolic oil, filtered	1,330	1,196
Hot-pressed naphthalene	594	893
Crude naphthalene		146
Flotation oil		18
Special tar products		515

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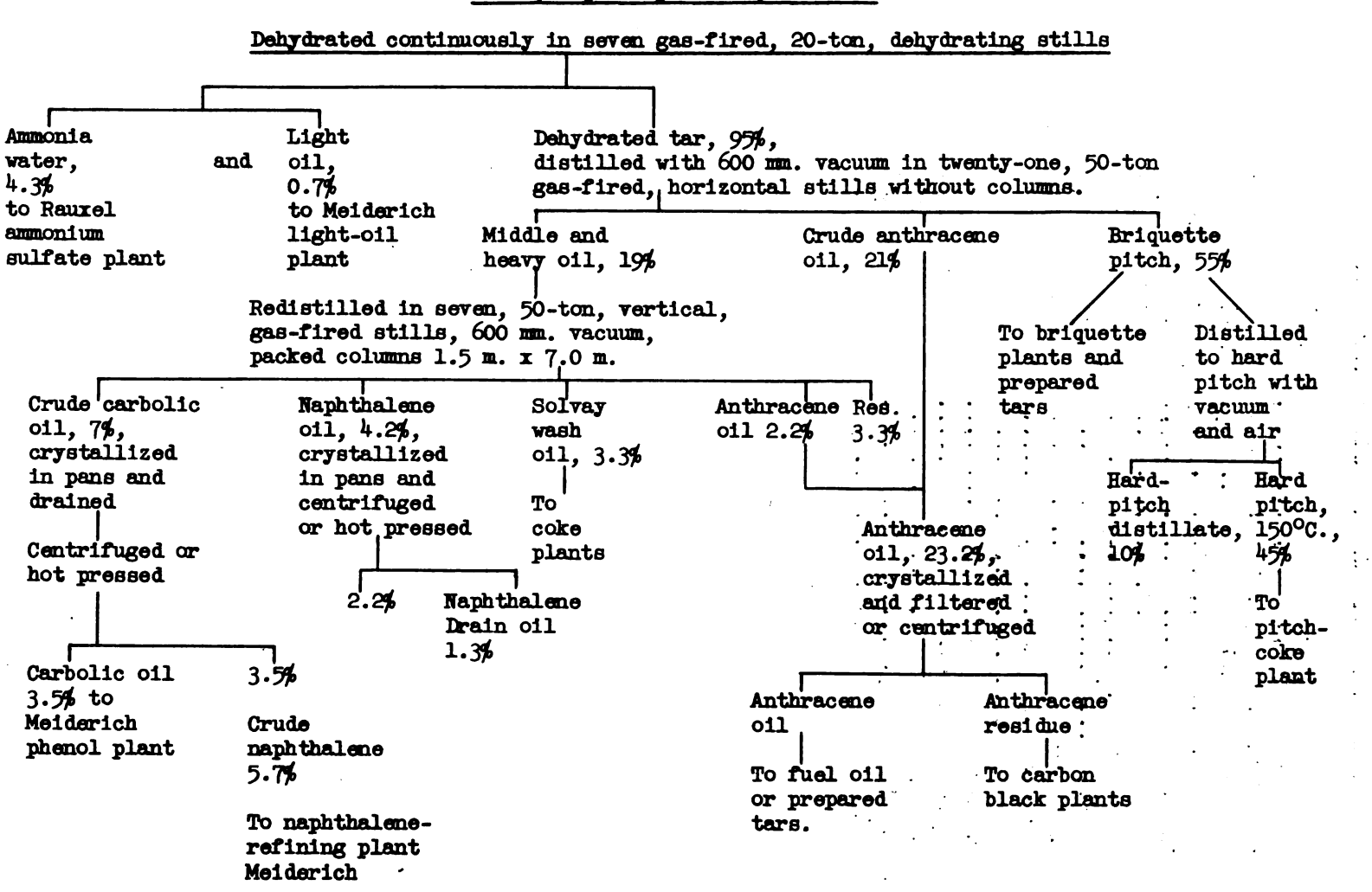
Crude, high-temperature, coal tar
Dehydrated continuously in seven gas-fired, 20-ton, dehydrating stills
Ammonia water, 4.3% to Raurel ammonium sulfate plant
and
Light oil, 0.7% to Meiderich light-oil plant
Dehydrated tar, 95%, distilled with 600 mm. vacuum in twenty-one, 50-ton gas-fired, horizontal stills without columns.
Middle and heavy oil, 19%
Crude anthracene oil, 21%
Briquette pitch, 55%
Redistilled in seven, 50-ton, vertical, gas-fired stills, 600 mm. vacuum, packed columns 1.5 m. x 7.0 m.
To briquette plants and prepared tars
Distilled to hard pitch with vacuum and air
Crude carbolic oil, 7%, crystallized in pans and drained
Centrifuged or hot pressed
Carbolic oil 3.5% to Meiderich phenol plant
3.5%
Crude naphthalene 5.7%
To naphthalene-refining plant Meiderich
Naphthalene oil, 4.2%, crystallized in pans and centrifuged or hot pressed
2.2%
Naphthalene Drain oil 1.3%
Solvay wash oil, 3.3%
To coke plants
Anthracene oil 2.2%
Res. 3.3%
Anthracene oil, 23.2%, crystallized and filtered or centrifuged
Anthracene oil
To fuel oil or prepared tars.
Anthracene residue
To carbon black plants
Hard-pitch distillate, 10%
Hard pitch, 150°C., 45%
To pitch-coke plant

Gesellschaft für Teerverwertung
Castrop-Rauxel

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New processes and products. - The only changes of any consequence made at this plant in recent years were as follows:

Hard-pitch manufacture. - In order to supply the new pitch coke plant of Verkaufsvereingung für Teererzeugnisse at Rauxel with hard pitch (150°C.), seven vertical 30-ton stills were installed. Each still was heated by gas and operated under vacuum (about 600 mm.) and agitated with air. The stills were charged with briquette pitch transferred directly from the tar stills, and during distillation of the briquette pitch to hard pitch, air was drawn through the pitch by the vacuum in the still. Distillation and air blowing was continued until the desired softening point was reached, at which time the temperature in the still was 380°-390°C. The high softening point pitch was then dropped by gravity into transfer tanks, from which it was loaded into tank cars by suction applied to the tank car. Transfer to the tank car by air pressure was tried at first, but the transfer lines became hot and, because of the danger of explosion, this method was discontinued. No difficulty was experienced in the use of the suction method of transfer.

Special pitch compounds. - To supply a recent demand for pitch compounds having better temperature susceptability characteristics than those of straight-run pitches, such compounds were made at Rauxel by fluxing hard pitch, made in the manner described above, with the distillate recovered from the same process, i.e., the distillation of briquette pitch to hard pitch. Hard pitch and hard-pitch distillate were mixed in a 36-cubic meter, steam-heated tank equipped for agitation with air.

Anthracene cooling and recovery. - The usual method of recovering anthracene and anthracene residue from anthracene oil has been to cool the oil in pans with manual stirring at intervals, then filter the slurry through vacuum filter pans and centrifuge the filter cake. However, during the war an improved process was installed, which consisted in cooling the anthracene oil in vertical water-cooled crystallizers with agitators and then centrifuging the crystalline slurry through Haubold vertical centrifuges. The anthracene residue produced in this manner contained less anthracene oil than that made in the usual manner. This method is described in greater detail in a report covering the Duisburg-Meiderich plant of this same organization.

Gesellschaft für Teerverwertung
Bochum-Gerthe

On September 20, 1945, the Bochum-Gerthe tar-distilling plant of Gesellschaft für Teerverwertung was visited to determine what changes, if any, had been made in manufacturing procedures or equipment in recent years. It was found that no important changes had been made.

This plant suffered very little damage by air raids or artillery. Damage was confined principally to the buildings (no equipment) of the anthracene and naphthalene hot-press plants. Normal operations of the plant were as follows:

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Tar dehydration. - About 120 tons of crude tar per day was dehydrated continuously in one vertical, 30-ton, gas-fired still. Water and light oil amounted to about 5 percent. The light oil (about 0.4 percent) was shipped to Meiderich for refining.

Tar distillation. - The dehydrated tar was distilled in eight 30-ton, vertical, gas-fired stills with 400 mm. vacuum. Temperature at finish was about 360°C. The fractions taken were: Middle oil, 200°-250°, 5 percent; naphthalene oil, 5 percent; heavy oil, 250°-300°, 8 percent; anthracene oil, 300°-350°C., 25 percent; briquette pitch, 54 percent.

Tar-acid extraction. - The middle oil was extracted with 18 percent caustic to remove tar acids, and the phenolate was sent to Meiderich. The neutral oil was combined with the heavy oil fraction (see above) and shipped to coke plants as wash oil.

The naphthalene fraction was cooled in pans, drained, and hot-pressed. The hot-pressed naphthalene was refined by washing at 90°-95°C. with caustic soda, sulfuric acid, formaldehyde (if available), and water. The washed naphthalene was redistilled.

Anthracene-residue recovery. - The anthracene oil fraction was cooled in pans and filter-pressed to remove anthracene residue, 4-5 percent, containing about 30 percent of oil. It was shipped to carbon-black plants. The filtered anthracene oil went partly to road tars but principally to fuel oil.

Manufacture of hard pitch. - The briquette pitch was transferred by suction to two vertical hard-pitch stills fired by gas, where it was distilled to hard pitch (150°C.) by hot flue gases (600°C.), heated, and made oxygen-free by direct combustion of coke-oven gas in the flue gas, using the oxygen contained in the flue gas to support the combustion. The temperature in the still at the end of distillation to hard pitch was about 380°C.

Hard-pitch cooling. - The hard pitch was cooled by means of an endless steel conveyor having a capacity of 50 tons per day. The pitch was transferred from the hard-pitch stills to two overhead feed tanks, from which it was discharged by gravity onto the steel conveyor. The latter was made up of overlapping steel plates mounted on rollers. From the upper end of the steel conveyor the pitch discharged into a car in the form of thin plates.

The plant was not operating at the time of the visit for lack of tar. Stocks of material on hand were 400 tons anthracene residue, 50 tons crude naphthalene, and 600 tons of hot-pressed naphthalene.

Rütgerswerke A.G., Castrop-Rauxel

The Castrop-Rauxel plant of Rütgerswerke was visited on September 17, 1945, and on two subsequent occasions. The manufacturing processes and products of this company were discussed with Dr. Carl Fehr, Dr. Geller, Dr. Carl Bellwinkel, and Dr. Paul Schurmann. The plant was inspected with some

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of these men. The quantity of tar processed in this plant in 1943 was 172,247 tons (metric).

About 40 percent of the equipment at Rauxel was destroyed during the war. One lime kiln in the tar-acid plant, a crude naphthalene plant having a capacity of 700 to 800 tons per month, a naphthalene-refining plant of 800 tons capacity, a crude anthracene plant of 120 to 140 tons per month capacity, pure anthracene equipment (30 to 35 tons per month), pure carbazole equipment and carbazole sublimation equipment (20 to 25 tons), and equipment capable of making 40 to 50 tons per month of acenaphthene, fluorene, and diphenylene oxide was destroyed. Also, the road tar-making and handling capacity was decreased 50 to 80 percent. The light-oil plant also was put out of commission. Very little damage was done to the batch tar stills and oil-redistilling stills with columns.

This plant distills dehydrated tar in vertical, gas-fired, 60-ton batch stills. Mounted directly above the stills are cast-iron fractionating columns 2 meters in diameter by 10 meters high. Each column has 40 bubble trays, and at the top is mounted a vertical-tube, water-cooled dephlegmator. The stills are operated under about 650 mm. vacuum. The following primary fractions are taken:

	Percent
Carbolic oil fraction	3.0
Naphthalene I (about 70°C.)	6.0
Naphthalene II (10°-15°C.)	6.0
Methyl naphthalene fraction	3.0
Acenaphthene fraction	3.0
Fluorene fraction	4.0
Phenanthrene fraction	3.0
Anthracene fraction (12-18 percent anth.)	10.0
Carbazole fraction	8.0
Briquette pitch, 70°-80°C.	50.0

Continuation of the distillation, with vacuum and some steam, yields the following products:

	Percent
Pyrene fraction (10-18 percent pyrene)	4.0
Chrysene fraction	4.0
Hard pitch (150°C.)	40.0

The accompanying table lists the products made at this plant during the first 10 months of 1944.

The newer developments at this plant that are worthy of note are the manufacture of Plastix, in special equipment, new apparatus for the cooling of pitch, and increased production of carbazole, acenaphthene, diphenylene oxide, pyrene, and symmetrical xylenol. The carbazole was employed by I. G. Farbenindustrie for the manufacture of tetranitro carbazole used in a grape-arbor insecticide. The acenaphthene and pyrene were used in

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making dyestuffs. The use of the diphenylene oxide is not definitely known, but it was said to have been used in scents. The symmetrical xylenol was shipped to Rütgerswerke at Niederau, where it was chlorinated for use in disinfectants.

Plastix manufacture. - For the manufacture of Plastix, a special plant was installed. It consists of a tank of 100 tons capacity heated by gas with ring burners, provided with distributing pipes for the introduction of air. A vapor off-take connects the tank with a water-cooled condenser. Following is a translation of a description of the process received at the Rauxel plant:

> The plastix oxidizing plant is charged with 70-80 tons of soft pitch with a melting point of 40°-50°C. The capacity of the oxidizing tank is approximately 100 tons, but there must be some empty space in order to avoid forming over into the distilling tube. In addition to soft pitch, naturally, other grades of any softening point may be used.
>
> The batch must be heated without interruption. When the temperature reaches 290°-300°C., oxidizing should be started. Oxidation should normally be finished after 10-14 hours. Approximately 400-600 cu.m. of air should be used per hour, regulating the pressure so as to insure thorough mixing of the mass.
>
> Oxidizing may be intensified by a small addition of steam. The oxidizing temperature must be raised to at least 350° and maintained at this level throughout the whole of the oxidizing operation. The use of heat is absolutely essential to achieve good results. Consumption of oxygen is controlled by analyzing the waste gas. Owing to the blowing with air and a little steam, roughly 5 to 10 percent distills off.
>
> When the mass has a softening point of 180°-200°C., the process is finished. Subsequently, the softening point is adjusted (in the same vessel) to the desired level by adding heavy tar oils, preferably free from residue.
>
> The quantity of oil required depends on the desired softening point, which generally is between 38° and 60°, although it is possible to keep the softening point at a higher level. The interval between the breaking point of the product and the softening point must be at least 40°C.

In addition to the above, it was stated that the best combination of oils for Plastix consists of 70 percent anthracene oil II, 350°-450°C. free from crystalline substances, and 30 percent of wash-oil fraction, 250°-320°C. The mixture should be free of crystalline substances. Approximately 50 percent of the above oil mixture is used to flux the hard pitch (180°-200°C.) produced in the manner described above.

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Another method for producing hard pitch in addition to the method for making Plastix was tried by Rütgerswerke several years ago. It was described in a British patent, No. 499,924, applied for on March 22, 1938. By this method, briquette pitch was heated to 300°-350°C. and blown with steam heated to 400°-500°C. The steam was superheated electrically. This process was tried on an experimental scale but was dropped because of the war. It was mentioned that the heat consumption of this process was high, but no attempt was made to conserve heat. The steam consumption was about 1-1/2 tons per ton of pitch. The pitch produced by the process was granulated with water.

Pitch cooling. - Considerable work has been done at this plant in recent years with a view to developing improved methods for cooling hard pitch and briquette pitch. The following method was used commercially at the Rauxel plant and also at the tar-distilling plant of Gutehoffnungshütte. It was especially suitable for cooling hard pitch. Liquid high softening-point pitch from an overhead feed tank flowed from small holes in the bottom of a horizontal pipe or manifold. The thin streams of molten pitch dropped onto water flowing in shallow, inclined troughs. The streams of pitch floated down the troughs with the water and cooled sufficiently before reaching the end to become semisolid. They then were separated from the water by an inclined grating made of iron bars spaced close together. While passing over the grating and dropping into a car, the moisture on the surface of the rods of pitch evaporated and the rods cooled sufficiently to break into small pieces.

The other method tried at Rauxel but not developed to the point of commercial use was intended primarily for the cooling of briquette pitch. The molten pitch flowed in a thin band onto water flowing down a shallow trough about 50 meters long. The nozzle by which the pitch was flowed onto the water was so placed that the pitch film remained on the surface for several meters. This arrangement allowed the pitch film to cool by air on the top while it was being cooled by water from below. It was claimed that by this arrangement the pitch became sufficiently solid so that water did not enter it when it became submerged in the water. A steel rod grating at the end of the trough separated the solid pitch from the water and discharged the granulated pitch into a car.

The water was recirculated.

The two methods of pitch cooling mentioned above have been patented in Germany by Rütgerswerke A.G. The patent numbers and dates are as follows:

No. 688,505, published Feb. 1, 1940.
No. 740,055, published Aug. 26, 1943.

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Shipments, 1944

	In kg.		In kg.
Acid resin	46,873	Flotation oil	128,586
Neutral light oil	41,520	Anthracene 40 percent	684,170
Soft pitch	2,017,292	Burning stock	7,195
Pitch	37,327,533	Anthracene 70 percent	15,015
Steel-mill tar	1,864,193	Test base A. T. (?)	20
Cable tar	249,570	Pyridine normal test	131,517
Prepared tar	1,058,593	Pure pyridine	21,950
Street tar or bond	391,641	Technical quinoline	13,463
Cold tar	160,330	High-boiling bases	20
Lithosote	1,231,341	Ammonium sulfate	357,792
Iron lacquer	697,422	Pyridine residue II	62,832
Insulation coating material	313,936	Pure anthracene	113,223
Straight 90 benzene	16,548	Sublimed anthracene	160
Motor benzene	1,057,602	Impregnating oil	146,070
Straight tuluol	177,252	Heating oil	3,693,006
Straight soluent benzene I.	1,688	Marine heating oil	27,569,801
Pure benzene	11	Solvay wash oil	6,867,641
Pseudo-cumene	1	Motor fuel oil	158,994
Pure xylol	567,587	Hydrogenated oil "M"	5,458
Neutral oil III	325,865	Hydrogenated oil I	11,096,885
Neutral oil IV	46,737	Hydrogenated oil V	8,139,072
Cumarone resin	1,167,936	Marine heating oil, medium pitch composition.	8,566,462
Benzoate liquor	179,490	Neutral oil	50,651
Phenol pitch	218,582	Xylene 1-2-4	5,781
Phenol/cresol mixture	277,510	Xylene 1-3-5	100,327
Phenol	2,766,519	Xylene 1-4-2	16
Liquid phenol	128,945	Hard pitch	1,392,604
Cresol DAB IV	2,152,801	Caminated pitch	27,265,885
Cresol DAB VI	1,373,070	Hard-pitch liquid	259,790
Pure ortho-cresol	273,026	Plastics	485,355
Pure meta-cresol	78,421	Cumarone hard residue	104,870
America-cresol	1,042,921	Ortho-oxy-diphenyl	9,210
Raw pyro-catechin	28,440	Para-oxy-diphenyl	35
Technical ortho-cresol	79,740	Special tar oil	1,225
Raw naphthalene	107,600	Technical fluorene	14,206
Warm pressed goods	2,137,830	Technically pure fluorene	3,485
Pure naphthalene	3,504,832	Phenanthrene, centrifuged	101
Filtered anthralene oil	1,067,152	Phenanthrene, untreated.	100
Carbolneum	3,485	Acenaphtene	229,384
Raw tar fatty oil I	450,240	Indene	13
Tar fatty oil provisions	50	Diphenyl oxide	235,911
Tar fatty oil II	175,937	Hydrocarbon mixture	9,982
Tar fatty oil III	143,443	Alpha-beta-methyl-naphthalene	22,683
Tar fatty oil IV	18,309		
Naphthalene wash oil	374,406		

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	In kg.
Alpha-methyl-naphthalene ...	3,751
Beta-methyl-naphthalene	315
Technically pure di-phenyl .	891
Carbazole 85 percent	59,676
Fluoranthrene	301
Pyrene	25,242
Chrysene	23
Raw ethylene chloride	123,680
Tar oil "Rutgers"	306
Straight soluent benzene II.	1,043,638
Pure toluene	223,915
Special beverage stock	981,040
Raw soluent benzene I	160
Raw America cresol	50,010
Pitch coke oil	30
	165,817,222

The production for 1944 covered only 10 months. From November 6, 1944, until the end of 1944, the factory was idle on account of the bombing on November 6, 1944.

Rütgerswerke Aktiengesellschaft, Berlin

Rütgerswerke Aktiengesellschaft, Berlin W-35, Lützowstrasse 33-36, was visited on October 31, 1945. The persons interviewed were directors Fabian, Müller, and Kahl and Professor Dr. Mallison. General information concerning the Rütgerswerke tar plants in eastern Germany was requested. The following information was obtained:

Erkner plant (near Berlin). - This plant is operating but at only about 10 percent of its capacity because of insufficient tar. The capacity of the plant is about 100,000 tons. The amount of tar distilled in 1943 was about 60,000 tons. The plant was not badly damaged during the war, and, except for the naphthalene-cooling and hot-pressing departments, could be operated at full capacity if enough tar were available.

The equipment and operating procedures at Erkner are like those used at Reurel by this same company. There are eight 40-ton vertical batch stills mounted directly below cast-iron columns for tar distillation, two continuous stills for tar dehydration (one damaged), two 100-ton and six 20-ton stills for benzol refining, a complete tar-acid plant (capacity about 10,000 tons per year) containing five crude-acid stills and one pure-phenol still, a naphthalene-cooling building and hot press (damaged), a cooling house for pure naphthalene (damaged), and plastix-making equipment (damaged).

In the way of new developments at Erkner, the following processes were mentioned:

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A method of making hard pitch (200°C.) from briquette pitch was tried, which consisted in passing briquette pitch heated to 300°-350°C. and steam heated to 400°-450°C. by means of an electric heater through a pipe into a flash chamber. The hard pitch coming from the flash chamber was granulated with water. According to Dr. Geller, who was interviewed later in regard to this process, steam consumption was about 1.5 tons per ton of pitch. The heat consumption was said to be high but might be improved by means of heat economizers. This process was tried on an experimental scale during the war but had to be dropped before development was completed.

In making plastix, it was said that the important thing is to use a proper combination of high- and low-boiling tar oils for fluxing hard (200°C.) pitch, and the quantity of oil mixture used must be just enough to "swell" the pitch and not dissolve it. The proportions used by Rütgerswerke were said to be about 30 percent oil and 70 percent pitch. The oil mixture contained oil distilling 380°-450°C. and oil distilling about 300°C.

In the benzol plant at Erkner, a method of removing thiophenes from benzol, to be used for synthetic phenol production, was used, which consisted in giving the benzol a preliminary wash with 66° sulfuric acid, then adding solvent naphtha II, containing indene, and washing again with 66° sulfuric acid. Agitation was continued for 2 to 4 hours. For benzene containing 0.2 to 0.4 percent thiophene, the amounts of acid and solvent naphtha II used were 14-18 percent by weight and 3 percent by weight, respectively. For a thiophene content of 0.05-0.15 percent, the percentages of acid and solvent were 6-12 percent and 1.5 percent, respectively. Following the acid treatment, the benzol was washed with water and soda.

Breslau. - The Rütgerswerke plant at Breslau was said to have been destroyed almost completely during the war.

Niederau. - The Rütgerswerke plant at Niederau (near Dresden), whose capacity was about 48,000 tons per year, is believed to have been dismantled and prepared for shipment to Russia.

Chemische Fabrik Weyl A.G., Mannheim

Chemische Fabrik Weyl A.G. at Mannheim-Waldhof was visited on December 1 and 4. The persons interviewed were Dr. Charles F. Lang (a director of Rütgerswerke and at present manager of the Mannheim plant) and Dr. Friedrich Cröy, director of Chemische Fabrik Weyl A.G.

This is the largest tar plant in the American zone of occupation. In 1943 approximately 73,000 tons of tar was processed. Crude-tar supplies came from coke plants in the Saar and from gas plants (about 50) in Hessen, Württemberg, and Baden.

About 15 percent of the equipment in this plant was damaged by air raids. The departments for cooling and hot-pressing naphthalene and for the production of fruit-tree carbolineum were destroyed. Some of the other departments were slightly damaged.

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Except for the stills and pitch-cooling pans, the equipment used at this plant is similar to that used in most of the German tar plants. The six stills, two of which are used for dehydrating and four for distilling tar, are heated by coal with mechanical stokers. Each still has a capacity of 62 tons and is constructed like a horizontal tubular boiler. 76 tubes, each 7,058 mm. long, 121 mm. O.D., and 108 mm. I.D., connect the two ends of the stills. The ends of the tubes extend through the end plates and are provided with a bead or rim, which keeps them from pulling away from the end plates when the stills are heated. Combustion gases pass through the tubes, and they are surrounded by the tar to be dehydrated or distilled. The two stills used for dehydration are not equipped with columns. The four tar-distilling stills have cast-iron bell-and-tray columns 15 meters high and 120 cm. in diameter. Each column has 48 trays and is provided with a water-cooled dephlegmator containing 17 vertical tubes, through which the vapors pass. The columns are so heated that reflux from them returns directly by gravity to the stills. The stills were originally installed by Weyl and are unlike those used by Rütgerswerke in its other plants, but the columns were installed by Rütgerswerke and follow their standard design.

Pitch cooling is carried out by equipment developed by Weyl, which operates satisfactorily but, according to the directors of the plant, is unnecessarily expensive. Eighteen shallow steel pans, each capable of holding one ton of pitch, are mounted in cascade arrangement above a railroad track and above a movable hopper, which can receive the pitch by gravity from any pan and drop it into a railroad car. Molten briquette pitch from a tank mounted above the topmost pan flows into the latter and overflows into each of the other pans until all eighteen are full. After cooling for a day, the pans are inverted by rotating them around trunnions mounted at each end. The solid pitch drops from each pan into the movable hopper, which conveys it to the car. The pitch-cooling building contains four units of this kind, or 72 pans divided into four groups of 18 each.

Following the destruction of the naphthalene hot presses, this plant began making 78° naphthalene by the redistillation of crystallized and drained naphthalene. A steam-heated vacuum still with columns is used for this purpose.

The various products made at this plant and their respective yields were as follows in 1943:

Water, 5 percent
Light oil, 2 percent, fractionated in benzol plant to produce crude benzol, toluol, xylol, and solvent naphtha. The first three were washed and redistilled to make pure light oil products and the solvent naphtha was shipped to Rütgerswerke at Rauxel.

Carbolic oil, 3 percent, shipped to Rauxel with the solvent naphtha.

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Naphthalene oil I, 7 percent, cooled in pans, drained, and hot-pressed to make 79.0°-79.5° naphthalene (or 78.0° to 78.5° a few years ago for American shipments). The oil from the hot presses was sent with the carbolic oil and solvent naphtha to Rauxel. The 79° naphthalene was washed and redistilled to make refined naphthalene. Present practice since the hot presses were destroyed is to crystallize this fraction, drain, centrifuge, wash, and redistill. The freezing point of the naphthalene is about 79.6°.

Wash oil, 7 percent, shipped to coke plants for recovery of light oil from the gas.

Anthracene oil I, 4 percent, shipped principally for fuel oil or hydrogenation. Small part for wood preservation (carbolineum).

Anthracene oil II, 7 percent, a part of this oil was crystallized in pans and filtered through vacuum filter pans to obtain anthracene residue for shipment to carbon-black plants. The filtrate went to fuel oil, naphthalene wash oil (used at coke plants), or carbolineum. Another part of anthracene oil II was mixed with wash oil or naphthalene oil, cooled in a water-cooled tank with agitation and centrifuged to make 40 percent anthracene. The centrifuge oil went to fuel oil.

Column oil, 2 percent, thin oil was drained from the column after completion of distillation and before filling the column with tar for the next distillation. The oil went to fuel or hydrogenati

Briquette pitch, 54 percent, cooled in 1-ton pans and shipped to briquetting plants.

When visited, this plant had received some crude tar from the Ruhr but was receiving virtually no tar from its regular sources of supply. Stocks on hand consisted of 7,000 tons of briquette pitch, about 300 tons fuel oil, 500 tons carbolic oil, and 300 tons of naphthalene.

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Gelsenkirchener Bergwerk Akt. Ges., Gruppe Bochum

Zeche Carolinenglück, Bochum

This plant was visited on September 20 with Dr. Geiselbrecht. The persons interviewed were works director Dipl. Ing. George Speckhardt and superintendent Voigt. They also conducted the trip through the plant and explained the various operations.

No part of the plant was working at the time of this visit, because permission to operate had not been granted. Some departments will require rebuilding before operation can be started, but others are ready for immediate use. The situation with respect to bomb damage is as follows:

Tar stills: Can be run at full capacity, i.e., 600 tons per day.

Oil-redistilling stills: Can be run at full capacity.

Naphthalene-washing equipment: Can be repaired.

Naphthalene-refining equipment: Can be used.

Phenol-extraction equipment: Can be repaired.

Naphthalene pans and hot presses: Totally destroyed.

Anthracene-recovery equipment: Totally destroyed.

Naphthalene-sublimation house: Totally destroyed.

The light-oil plant, although severely damaged, can be placed in operation soon, because the equipment is in good condition. Most of the damage suffered by this plant was confined to the building.

When running at full capacity, this plant received 600 tons of tar per day from eight sources, namely: Carolinenglück, Bruchstrasse, Donnenbaum, Nordstern, Alma, Friedrich Wilhelm Hütte, Hoerderverein, and Tremonia. The tar was received by tank cars and unloaded by dropping it into troughs beside the track that run to tar-storage tanks.

Dehydration. - Dehydration of the crude tar was carried out in one 50-ton, vertical, pot-type still running continuously without column. The water and light oil together amounted to about 5 percent.

Distillation. - Seven stills were used for distilling the crude tar. They are divided into two groups. Three 50-ton, vertical, pot-style stills without columns are in one group, and four 50-ton horizontal stills provided with two fractionating columns are in another group.

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The horizontal stills have two large heating tubes (as at Gutehoffnungshütte). They are so connected with the columns that any one of the four stills can be run with either of the columns. Each of the columns is 2 meters in diameter and 19 meters high. There are 19 sections and 38 trays, the tray spacing being 500 mm. They are provided with water-cooled dephlegmators, condensers, and barometric legs for the condensates. The columns are not enclosed. Both the vertical and horizontal stills operate under about 650 mm. vacuum.

The products from the vertical stills were:

Water and light oil, 5 percent - The light oil (about 2 percent) went to the benzol plant.

Crude wash oil, 15 percent - Redistilled in four vertical stills with small bell and tray columns (not enclosed) to make:

Carbolic oil shipped to Meiderich for tar-acid extraction.

Naphthalene oil washed with 20 percent NaOH to recover tar acids for shipment to Meiderich. The neutral oil went to wash oil or fuel oil.

Wash oil - Shipped to coke plants for light-oil recovery.

Residue - Returned to crude tar.

Anthracene fraction, 17 percent - Crystallized in pans, drained, filtered (with vacuum filter pens), and centrifuged to obtain anthracene residue (20 percent anth.) for carbon black or refining to 40 percent anthracene and anthracene oil (to road tars or other prepared tars or fuel).

Heavy oil, 8 percent - Hydrogenated or mixed with bitumen for roads.

Hard pitch, 50 percent - Shipped to pitch coke plant at Lothringen.

From the horizontal stills with columns, the following products were obtained:

Carbolic oil, 4 percent - Shipped to Meiderich.

Naphthalene fraction, 8-9 percent - 65° freezing point, washed for tar acids (phenolate to Meiderich), crystallized in pans, drained, and hot-pressed to make 78° + naphthalene and wash oil.

Wash oil, 8 percent - Used directly for light-oil scrubbing.

Anthracene oil, 20-22 percent - Cooled in pans, filtered, and centrifuged.

Briquette pitch - Shipped to coal-briquetting plants.

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The most interesting points observed at this plant were the following:

Oil-redistilling stills. - (Four horizontal stills equipped with two effective 38-plate fractionating columns.) This installation (described above) was said to have been built by Carl Still of Recklinghausen.

Tar acid-oil extraction. - Two vertical cone-bottom tanks, which previously had been employed as benzol washers, were being used as extractors for the removal of tar acids from tar-acid oil. Each tank is filled about one-half to two-thirds full with 20 percent caustic-soda solution. Then the tar acid oil is pumped continuously into the tank through a pipe that extends almost to the bottom of the cone. The oil rises through the caustic-soda solution and overflows near the top of the tank into a run-down tank. The tar acids are extracted from the oil as it rises through the caustic soda. Little or no emulsification takes place, because agitation is not employed. When the caustic becomes saturated with tar acids, it is replaced with fresh caustic, and the cresylate is sent to Meiderich. The process was developed by the director of the plant, Dir. Ing. George Speckhardt. He provided a copy of his patent application in which the process is described.

Pitch cooling. - Two methods of cooling pitch have been used at this plant. One is an inclined conveyor with overlapping plates. The molten pitch is fed onto the lower end of the conveyor, and plates of solid pitch are discharged from the upper end into a railway car. According to Mr. Speckhardt, this type of cooler is suitable only for hard pitch (for briquette pitch its capacity is low), and maintenance costs are considerable. The method which Mr. Speckhardt favors, for either briquette or hard pitch, makes use of an elevated tank for precooling the pitch, a feed drum with several valves and small tubes on the bottom, a rectangular water tank the bottom portion of which is arranged with sloping sides, and a screw conveyor and perforated bucket elevator discharging into railway cars. To operate this device, the liquid pitch to be cooled is pumped to the overhead precooling tank, where it is cooled atmospherically to below 300°C. (about 230°-235°) for hard pitch and below 150° (about 120°C.) for briquette pitch. It is then run into the feed tank, and the valves are opened to permit the pitch to run through the bottom openings into the water. The streams of pitch congeal in the water and drop to the bottom of the water tank, where they are moved by the screw conveyor to the end of the tank. They are then transported by the inclined perforated bucket elevator to a chute, which delivers them in granulated form into an open railway car. This arrangement for the cooling of pitch is described in German Patent 743678, issued to George Speckhardt, works director of the Carolinenglück plant.

The benzol plant building at this plant had been heavily damaged by air raids, but the equipment was not appreciably affected and could be placed in operation again in a rather short time.

Stocks of raw and finished materials on hand at this tar plant were not large. The quantity of crude tar in storage was only 400 tons.

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Gelsenkirchener Bergwerks A.G.
Chemische Betriebe Pluto-Wilhelm, Wanne-Eickel

The date of the visit to this plant was September 15, 1945. Works manager Slumberger was interviewed.

The plant is divided into the following departments, with separate buildings for each: (1) Tar distillation, (2) cements and coatings, (3) bituminous paints, (4) lubricating oils and greases, (5) reclaiming of used lubricating oils.

The tar-distilling plant is small and of the type most frequently used in Germany. It processed approximately 2,300 tons of crude tar per month and 400 tons of benzol wash oil and coumarone residues. There were eight vertical gas-fired stills of 20 tons capacity each. Some of the stills and all of the condensers and receivers were damaged or destroyed by bombs. One still with a temporary condenser was operating at the time of the visit. The stills were operated under vacuum to briquette pitch and also to hard pitch (100°C.+). The following fractions were collected from dehydrated tar: (Dehydration continuous in a separate still.)

		Percent
1.	Water and light oil	0.5
2.	Middle oil, 200°-235°	10-12
3.	Heavy oil, 235°-250°	3-5
4.	Anthracene oil (light), 250°-270°	8-10
5.	Anthracene oil (med. heavy), 270°-300°..	5
6.	Anthracene oil (heavy), 300°-(360°-400°)	3-5
7.	Pitch, 100°-150° K.S.	45-50

The fractions were handled as follows:

1. Light oil used in bituminous paints.

2. Middle oil cooled in pans and drained, 55°-60°.
   Naphthalene centrifuged or sent to carbon-black plants or fuel oil.
   Drain oil redistilled to make wash oil, coal-flotation oil, or oil for shipment to tar-acid plants.

3. Heavy oil cooled in pans. Naphthalene added to that from (2). Drain oil to wash oil or carbolineum.

4. Cooled in pans, filter-pressed; oil used as flux for road tars, steel-works tar, refined tar, or as fuel. Anthracene residue (2.0-3.5 percent) shipped to carbon-black plants.

5. Cooled in pans, filter-pressed: Anthracene residue added to that from (4); oil redistilled to make light and heavy fluxes for special purposes.

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6. Used in making lubricating oils after cooling, filtering and redistilling. Also used for flux for roofing and waterproofing compounds.

7. Hard pitch, shipped to pitch coke plants.

This tar plant specializes in making compounds of the type called Plastix in Germany. Hard pitch is fluxed with heavy anthracene oil or a mixture of the latter with hard pitch distillate. Such mixtures have improved temperature characteristics and are used in roofing, waterproofing, coating, and paint compounds.

The plant for making cements and coatings is equipped with three horizontal heavy-duty mixers and two smaller pony mixers for making filled compounds. Soft pitch is mixed with various fillers and in different amounts to make hot application compounds for use on roofs, foundations, tunnels, cables, pavements, etc.

The factory for making bituminous paints has five steam-jacketed, agitated, vertical mixing tanks with reflux condensers. Coal-tar pitches and petroleum asphalts are fluxed with solvents to make metal paints of all kinds.

The lubricating-oil and grease factory is well-equipped with mixers, coolers, and kneaders for making various kinds of lubricants and greases. During the war, heavy anthracene oil was used as a substitute for petroleum oil in the formulation of lubricants, greases, and puttys. The products were used for heavy-duty purposes, for which they were considered to be satisfactory.

The plant for reclaiming used lubricating oil settled, dehydrated, and centrifuged such oils and returned them to the plants from which they were received.

Bergbau A.G., Ewald-König Ludwig, Herten, Germany

Tar-Distillation Plant

The König Ludwig tar-distilling plant, near Recklinghausen, was visited by the writer, Mr. Hunter (civilian officer, Military Government, Gelsenkircken), and Dr. Geiselbrecht on September 27.

The plant officials who conducted the inspection were Westmann (director of coke plant and tar plant), Flottmann, assistant director of coke plant and tar plant, Ahnen, assistant superintendent of coke plant, and Klussmeier, foreman of tar plant.

The plant had not been damaged by bombs or artillery fire, and operations could be resumed immediately if permission to reopen the plant were received.

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Operations were stopped completely on March 21, 1945. Since that time tar has been received at the rate of about 300 tons per month from König Ludwig mine, four-fifths which is carbonizing 300 tons of coal per day. This is half of the plant's carbonizing capacity. The amount of coal mined per day at this plant is now 3,000 tons. Ordinarily it would be 6,000 tons, but destruction of one of the two hoisting drums cut production in half.

The tar plant has on hand at this time about 2,000 tons of crude tar, 385 tons of anthracene residue, 210 tons hot-pressed naphthalene, 5,000 tons briquette pitch, 200 tons hard pitch, 272 tons crude naphthalene, 25 tons naphthalene for naphthalene motors, 68 tons of 40 percent anthracene, and about 1,500 tons of various grades of oil.

Eighty-one people were employed during the war. No operators are employed at this time while the plant is shut down. All of the equipment in this plant is well-arranged and in good condition.

Tar Receiving

Tar arrives at the plant in tank cars, which are switched into a large building with two tracks running lengthwise. The building is long enough to accommodate about 10 cars on each track. Between the two tracks in a steel trough into which the tar from cars on either track can be discharged by gravity. The trough delivers the tar to a sump, from which it is pumped to storage. It was explained that this tar-receiving building was provided to take care of shipments of brown coal tar, which König Ludwig expected to handle because another plant that had been distilling this tar had been destroyed. Brown coal tar becomes more viscous than bituminous coal tar on cooling, and the building was provided to keep the tar warm.

Tar Dehydration

Two 50-ton, vertical, gas-fired stills are used for continuous dehydration of the tar. The crude tar is pumped through a vapor-to-tar heat exchanger or condenser and to an overhead feed tank, from which it flows to the two stills. The dehydrated tar flows continuously to receivers, from which it is pumped to storage, and the vapors pass through tar-cooled and water-cooled condensers. The combined condensate is decanted continuously to separate light oil (about 1 percent) and water. The light oil is sent to the benzol plant for refining.

Tar Distillation

The dehydrated tar is distilled to briquette pitch in four gas-fired horizontal stills of 50 tons capacity each. They are constructed like those used by Carolinenglück and Gutehofnungshütte. The heat passes first through two tubes, then back around the lower half of the still, and returns to the stack through a horizontal flue located about 2 feet below the bottom of the still. The stills are not equipped with columns. The only fractions taken to briquette pitch are about 24 percent "creosote" and about 18 percent anthracene oil.

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Pitch Distillation

In addition to briquette pitch, this plant makes hard pitch, about 150°C. K and S, for electrode-coke production. The briquette pitch to be run to hard pitch is transferred from the four horizontal stills described above to three gas-fired vertical stills, one of 50 tons capacity and the other two of 30 tons capacity each. The 50-ton still is sometimes employed to distill dehydrated tar all the way to hard pitch. The distillation to briquette pitch, either in the horizontal stills or in the 50-ton vertical still, is carried out with vacuum and no agitation, but the distillation from briquette pitch to hard pitch in the three vertical stills is effected by blowing the pitch with air. Some heating is done at the same time. The air with which the pitch is agitated is introduced under 4 to 5 atmospheres. The amount of air used was said to be small, but the quantity was not stated. The time required to go from briquette pitch to hard pitch is 4 to 5 hours.

Pitch Cooling

Briquette pitch is cooled entirely in steel pans. The pan building has 20 pans of 70 tons capacity each mounted on a single level above railroad tracks. An arched roof with central ventilator extends over all the pans. Pitch is discharged from the pans into the cars below through narrow chutes arranged like funnels. Fifteen of these pans were used ordinarily for briquette pitch. The other five were used for cooling the hard pitch that was shipped to the pitch-coke plant at Lothringen.

Another method of cooling was used for the hard pitch that was added to special low-ash coal (30 percent hard pitch and 70 percent special coal) for the production of low-ash coke in byproduct coke ovens. The method of cooling was as follows:

A steel drum about 15 feet long and 30 inches in diameter revolves about 30 times per minute in a horizontal position below a liquid, hard pitch-distributing manifold and a water-distributing manifold. The water falls on the drum a short distance ahead of the liquid pitch, so that the latter actually falls onto a film of water. The surface of the drum is divided into channels about 4 inches wide by means of rings about 1/2 inch high and 1/2 inch wide. A doctor blade pressed against the bottom of each channel removes the thin film of pitch that has been solidified by the thin film of water on which it was deposited, and the flake pitch and water fall from the blades onto water flowing in shallow troughs. The troughs discharge onto a grating made of steel bars placed close enough together to keep the pitch flakes from falling through but with sufficient clearance to drain the water into a tank below, from which it flows through a coke bed to another tank. From there it is pumped back to the chilling roll. The flake pitch slides down the grating and onto a belt conveyor, which transports it to a car. A fume hood mounted above the roll conducts objectionable vapors away from the equipment. This chilling device was said to have a capacity of 20 tons per hour when operating on hard pitch. This appears to be a practical pitch-cooling device of large capacity.

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Oil Redistillation

As stated above, the oil fractions taken when making briquette pitch are 24 percent "creosote" and 18 percent anthracene oil. The creosote is redistilled to make 0.5 percent light oil, 3.0 percent carbolic oil, 10.0 percent naphthalene oil, 5 percent wash oil and 5 percent residue (all percentages on the basis of dehydrated tar). Oil redistillation is carried out in six 40-ton, vertical, gas-heated stills equipped with fractionating columns. Each column is 1.2 m. in diameter by 7 m. high (over-all) and provided with 12 trays and bubble caps. The top of each column is a four-plate dephlegmator. Reflux is controlled by regulation of the cooling water to the dephlegmators.

Naphthalene

The naphthalene-cooling building has horizontal-drain oil tanks on the first floor, anthracene-cooling pans on the second floor, and naphthalene-cooling pans on the third and fourth floors. Each naphthalene floor has 10 pans about 15 by 15 by 4 feet arranged in two rows of five each. Between the pans on the first naphthalene floor (No. 3) is a screw conveyor, into which the naphthalene may be shoveled from the pans on that floor. Between the two rows of pans on the fourth floor are chutes, which discharge into the conveyor on the third floor. The anthracene pans are drained by gravity to tanks on the first floor. By means of a conveyor, the naphthalene from the pans (about 69°C.) is transported to the hot-press building. It goes into hoppers above two hot presses, drops into their mixers, where it is mixed at a temperature of about 30°C. (said to be no admixture of molten naphthalene or oil at this plant) and pressed at that temperature.

Anthracene

The anthracene oil from the primary distillation to briquette pitch (without redistillation) is crystallized in the pans mentioned above and in another anthracene cooling house with pans. After crystallation it is pumped to vacuum pan filters (nütchen) mounted above filtrate receiving tanks. The vacuum filters are rectangular pans about 10 by 15 feet and are not equipped with agitators. The anthracene residue, when removed from the filters, is conveyed to a covered storage outside the filter-pan building.

To make 40 percent anthracene, the anthracene made as described above is dissolved in an equal quantity of wash oil in a horizontal steam-heated tank, recrystallized, and centrifuged continuously with two Hauboldt centrifuges. The amount of 40 percent anthracene made is not large. The centrifuges and dissolving equipment are in the same building as the vacuum filter pans.

Naphthalene Briquettes

In a separate building is housed the equipment used for making naphthalene briquettes for stationary engines. Drained naphthalene from the pans

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mentioned previously is recrystallized in elevated pans, drained, crushed, and molded into blocks about 3 by 3 by 2 inches. The two presses used for this purpose were made by Fritz Kalian, Berlin-Lichtenberg, Herzbergstr. 102-104. They operate in the same manner as ball presses.

There were two or three ball presses in this same building, as well as equipment (not used) intended for the continuous extraction of tar acids from brown coal-tar oil.

Special Products

A separate building housed mixing and barreling equipment for heavy greases. A petroleum lubricant was compounded with soap to make heavy greases for use by the König Ludwig mines. No tar oils were used in these products.

This plant did not make special pitch compounds like plastix.

Gutehoffnungshütte, Zeche Sterkrade near Oberhausen-Sterkrade

The Sterkrade plant of Gutehoffnungshütte was visited on September 19, 1945. The persons interviewed were Kokerei Director Dr. Kleingrotthaus and Kokerei Betriebsführer Ludwig Koerdt.

The plant had been damaged by bombs and artillery to the following extent: A naphthalene-cooling and hot-press building was destroyed by incendiary bombs, and one oil-redistilling still was damaged by artillery.

Stocks of material on hand were 400 tons of crude tar, about 3,500 tons of pitch, and about 400 tons of tar oils. Tar receipts from the Jacobi mine amounted to about 50 tons per day.

Crude tar distilled by this plant during the period 1941-43 amounted to about 73,000 tons per year. Products made were heavy solvent, carbolic oil, crude naphthalene, wash oil, anthracene residue, anthracene oil, hard-pitch distillate, briquette pitch, and hard pitch. The operating procedure is illustrated by the accompanying flow diagram.

Briquette pitch is cooled in pans, but the hard pitch is cooled in a special device in which the molten pitch, at 250°C., flows from 25 small tubes, about 10 mm. in diameter, onto the surface of water flowing in small semicircular troughs about 10 cm. wide and 7 m. long. The streams of pitch, congealed by the water, flow with the latter to the ends of the troughs, where they are separated from the water by a grating. They fall into a car, which transports them to pitch-coke plants. The cooling water drops into a tank and is recirculated to the troughs. The temperature of the water is about 40°C. The quantity of water used per hour is about 20 to 30 cubic meters. The amount of pitch cooled per hour is about 5 to 8 tons.

For the making of blended mixtures such as road tars and special pitch compounds, two horizontal tanks equipped with mechanical agitators are employed.

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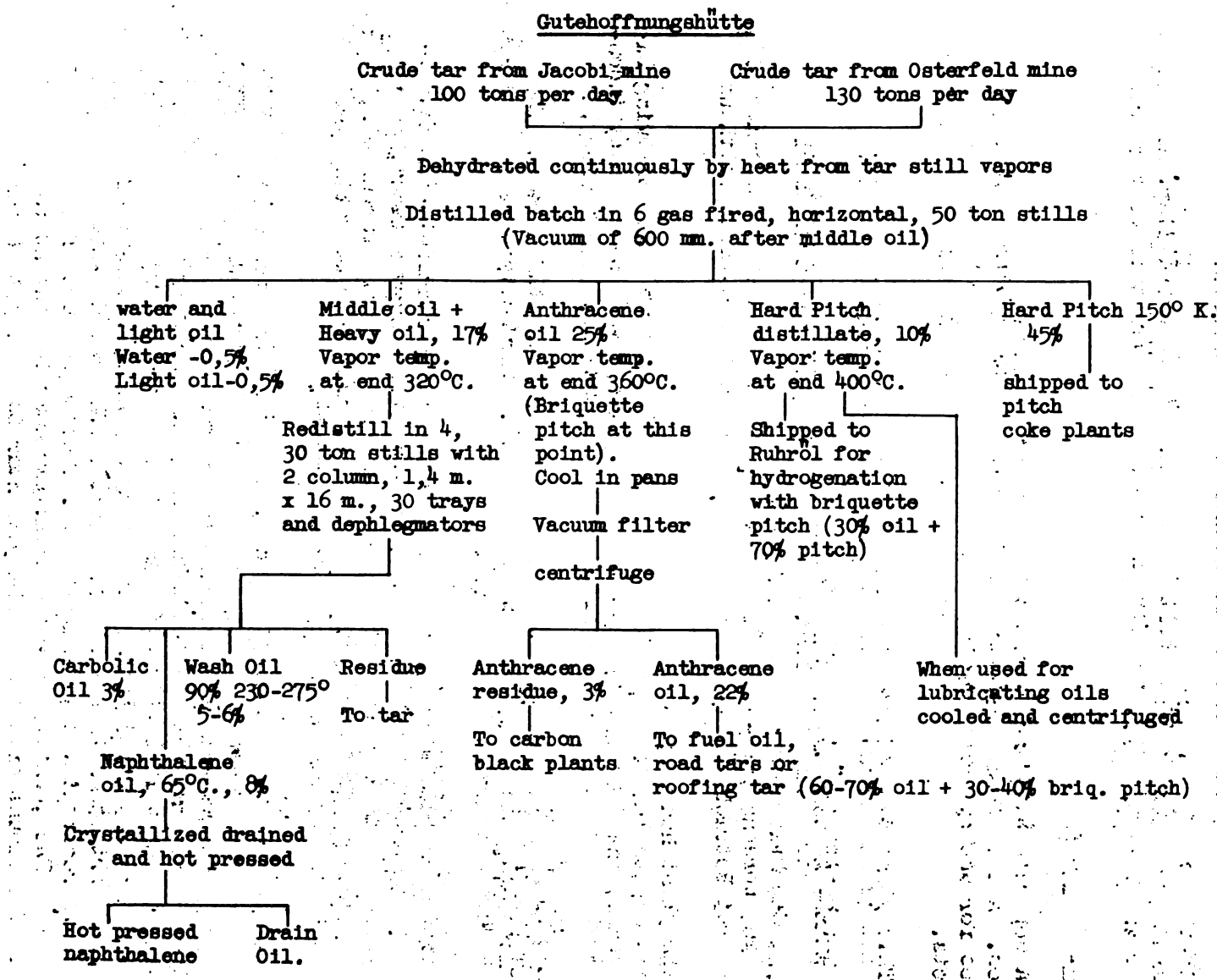
Gutehoffnungshütte
Crude tar from Jacobi mine 100 tons per day
Crude tar from Osterfeld mine 130 tons per day
Dehydrated continuously by heat from tar still vapors
Distilled batch in 6 gas fired, horizontal, 50 ton stills (Vacuum of 600 mm. after middle oil)
water and light oil Water -0,5% Light oil-0,5%
Middle oil + Heavy oil, 17% Vapor temp. at end 320°C.
Redistill in 4, 30 ton stills with 2 column, 1,4 m. x 16 m., 30 trays and dephlegmators
Anthracene oil 25% Vapor temp. at end 360°C. (Briquette pitch at this point).
Cool in pans
Vacuum filter
centrifuge
Hard Pitch distillate, 10% Vapor temp. at end 400°C.
Shipped to Ruhröl for hydrogenation with briquette pitch (30% oil + 70% pitch)
Hard Pitch 150° K.S. 45%
shipped to pitch coke plants
Carbolic Oil 3%
Wash Oil 90% 230-275° 5-6%
Residue
To tar
Naphthalene oil, 65°C., 8%
Crystallized drained and hot pressed
Hot pressed naphthalene
Drain Oil.
Anthracene residue, 3%
To carbon black plants
Anthracene oil, 22%
To fuel oil, road tars or roofing tar (60-70% oil + 30-40% briq. pitch)
When used for lubricating oils cooled and centrifuged

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Teerdestillation Mathias Stinnes, Essen-Karnap

Teerdestillation Mathias Stinnes 1/2/3, Essen-Karnap, was investigated on September 21, 1945. The persons interviewed were Berwerk's Direktor Dr. Broche, Betriebsführer Halfmann, and Betrieb's Direktor Rummel.

War damage to this plant was very light, and all departments could be operated. The plant was operating at partial capacity when visited (50 tons per day).

This well-arranged and well-operated tar plant distilled about 38,000 tons of crude tar per year during the period 1941-43, but in addition it distilled a residue coming from the hydrogenation plant of Ruhrol A.G. Bottrop, an associated company. The quantities of crude tar and hydrogenation residue distilled per day averaged about 100 tons and 140 tons, respectively, during the war years.

If distilled by itself, the hydrogenation residue would yield about 10 percent heavy oil and 90 percent briquette pitch, but actually the hydrogenation residue was mixed with crude tar in the above proportions, so the heavy oil from the hydrogenation residue was combined with that from the tar, and the briquette pitch was a mixture of pitches from the two sources.

Because the briquette pitch was of mixed origin, this company carried on considerable investigational work to determine and establish its value for coal briquettes. It approximated but did not quite equal in binding capacity briquette pitch from tar alone.

The operations at this tar plant are carried out in the following equipment:

Dehydration. - Tar is dehydrated by using it to condense the vapors from the tar stills.

Distillation. - Six horizontal 50-ton stills heated by gas through two large tubes in each still with return of combustion gases beneath the still before passing to stack.

Oil redistillation. - Two vertical 25-ton stills equipped with 800 mm. diameter bell and tray columns, and two vertical 50-ton stills with 25 tray columns 1,200 mm. diameter and 12 m. high.

Pitch cooling. - Steel pans mounted above railroad tracks.

Naphthalene recovery. - Cooling pans, centrifuge, and hot presses.

Tar-acid extraction. - Batch extraction in tank with mechanical agitator.

The following are the operations and yields of products at this plant when crude tar alone is worked up. When the mixture of tar and hydrogenation residue was processed, the yields of pitch and anthracene oil were increased and the yields of all other products were decreased.

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Tar dehydration:

	Percent
Water	4.0
Light oil	0.5

Tar distillation:

Middle oil, 25 percent, redistilled light oil	0.5
Naphthalene oil, 15 percent, cooled in pans, drained and centrifuged or hot-pressed crude naphthalene	8.0
Naphthalene drain oil, 7 percent, extracted with crude caustic soda:	
Sodium phenolate (as phenols)	0.5
Neutral oil	6.5
Wash oil	6.0
Anthracene oil, 20 percent, filter-pressed:	
Crude anthracene	5.0
Filtered anthracene oil	19.0
Briquette pitch, 70° softening point, K.S.	50.0

Hanseatische Teerprodukte Fabrik, Haltermann & Co., Hamburg, Germany

This plant was inspected on October 10, 1945. Johann Haltermann, owner, and Dr. Alfred Glawe, director.

It is an old tar-distilling plant, but it is the largest and therefore the most important plant of its kind in northern Germany. It is distilled about 30,000 to 35,000 tons of tar per year during the war. The tar was received from city gas works and also from byproduct coke-oven plants.

In addition to the tar-distillation plant, this company has a benzol-refining plant in the same location. Bomb damage during the various air raids was large. Mr. Haltermann and Dr. Glawe estimated that about 65 percent of the plant had been damaged by bombs. The naphthalene-refining plant was totally destroyed; about 45 storage tanks out of more than 60 were destroyed or badly damaged; the settings of the tar stills were broken and are now being rebuilt, and considerable damage was suffered by several of the buildings.

At the time of this visit one tar still was operating for the production of briquette pitch and various oil fractions.

A new 50-ton oil-redistillation still and column is being installed, and preparations have been made to install a continuous dehydrating still.

Products.

The products made in the plant were as follows:

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Light oil products - benzol, toluol, xylol, solvent naphtha; naphthalene products - hot-pressed naphthalene, refined naphthalene, balls, flakes, and naphthalene oil for insecticides; anthracene residue for carbon-black manufacture; anthracene oil for fuel, road tars, and refined pitches; crude, sprung, tar acids shipped to Meiderich and Rauxel; wash oil of regular grade and special wash oil filtered at 0°C. (for use at coke plants in naphthalene scrubbers); briquette pitch; road tars; and roof cement.

Manufacturing Procedures and Equipment.

The crude tar was dehydrated and distilled in two 25-ton vertical batch stills and two 40-ton vertical batch stills. The stills were operated under approximately 600 mm. vacuum. The primary fractions were: Water, 5.0 percent, and light oil, 0.2 percent; middle oil, 10 percent, anthracene oil, 20 percent, and briquette pitch, 55 percent (all yields approximate). The light oil was worked up in the benzol-refining plant together with light oil brought in from coke-oven plants (about 6,000 tons per year). Steam-heated stills equipped with bell and tray columns were used for this purpose. One still, used especially for the fractionation of toluene, has two columns in series. It was said to be very satisfactory. The fractionation and washing operations carried out in this plant were like those of other plants of the same kind. Disposal of the benzol sludge resulting from the sulfuric washing of various light oils was carried out as follows: two parts of sludge were mixed with one part light oil (boiling range 200°-250°C.) and, while mixing, additional water was added. The mix was then allowed to settle, after which the dilute sulfuric acid was drained to the sewer and the oil layer was washed with a solution of sodium carbonate. After settling again, the sodium carbonate solution was drained to the sewer and the oil layer was pumped to crude-tar storage.

The medium oil resulting from the primary distillation was redistilled in a 50-ton vertical still upon which was superimposed a 40-tray column 2 meters in diameter. There are 42 bells 200 mm. in diameter on each tray. The still was operated under 65-70 cm. vacuum. The fractions collected were:

Crude benzol. - Sent to light-oil plant.

Carbolic oil. - Washed with NaOH and then redistilled for additional benzol and solvents in the light-oil plant. Phenolate sprung with H_2SO_4. Sodium sulfate to sewer and the crude phenols to Rütgerswerke at Rauxel.

Naphthalene oil. - Cooled in pans and drained, hot-pressed, washed (in two lead-lined washers), redistilled, and made into balls, flakes, etc. The naphthalene oil was sold to makers of creolin (Pierson) and insecticides (Altenlander Pflanzenschutzmittel Fabrik and Staedter, Holland, and Stade). The naphthalene washing was carried out with six successive washes with

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66°Be H_2SO_4, two washes with 10 percent NaOH, two water washes, and a final wash with 10 percent NaOH. Washing was followed by a final redistillation in a steam-heated still under vacuum and fractionated with a bell and tray column. The naphthalene sludge was disposed of in the following manner: All of the washes (H_2SO_4, NaOH, and H_2O) from the washing operation described above were collected together in an acid-proof, brick-lined, rectangular, concrete tank. More water was added (about 10 parts water to one of sludge). After settling, the supernatant aqueous portion was run to the sewer (Dr. Glawe said it was virtually neutral), and the mixture of naphthalene and resins was shoveled from the tank and burned under the boilers.

Wash oil. - A part was sent directly to coke plants for light-oil recovery, and the remainder was refrigerated to 0°C., filter-pressed, and shipped to coke plants for scrubbing naphthalene from the gas.

Residue from oil-redistillation. - Added to filtered anthracene oil. Anthracene oil from the primary distillation was cooled in pans filtered in vacuum filters, and used in road tars or for making prepared tars. The anthracene residue, filtered from the anthracene oil was sold to the carbon-black plants.

A special roof cement was made for one customer. The composition of this material was as follows:

	Percent
Coal-tar pitch, 20°C. K&S	53-79
Heavy benzol, 160°-200°	6-7
Asbestos fiber)	15-40
Kieselguhr)	

The composition of the cement was varied to suit climactic conditions. It was used as a heavy roof-coating material.

In addition to the equipment discussed above for the manufacture of tar products, this company has a separate department for redistilling a light petroleum distillate imported from America. From it are made gasoline and various solvents for the rubber, paint, chemical, vegetable-oil, and other industries. Steam-heated stills provided with effective fractionating columns are used for this purpose. The columns are bell and tray with pump-back reflux. (All other columns in the plant use dephlegmators.)

Mr. Haltermann commented on the fact that the wooden roofs on the benzol-refining and benzine-refining buildings had proven to be superior to

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steel roofs during the air raids. They burned and fell on the stills, but they did not collapse the walls (as steel roofs did in other cases), and the stills and receivers (although full of flammable materials) did not ignite because they were heavily insulated.

Aktiengesellschaft Johannes Jeserich, Hamburg, Germany

Tar Distillation and Manufacture of Bituminous and Resinous Paints and Coatings

The plant of Aktiengesellschaft Johannes Jeserich, Zweiniederlassung, Hamburg, Germany, was visited on October 13, 1945, to find out what products are made by this company from coal tar.

The only person interviewed was Johannes Jeserich, the owner of the plant, who took the writer through the plant and explained its operations.

The plant had been heavily damaged by one bombing attack of about 10 minutes' duration. The roofing-felt plant, warehouse, and other buildings were completely destroyed. The tar-receiving equipment, tar-storage tanks, and tar stills were not damaged appreciably and are in operation. Also, the laboratory building (used also as an office building), the plant formerly used for distilling wood distillates, and a small varnish-cooking plant were largely undamaged and are still usable.

There were virtually no stocks of raw materials or finished materials on hand at this plant, and Mr. Jeserich was anxious to obtain more raw materials so that he might increase those operations that can be carried out with the remaining equipment.

Crude coke-oven tar is distilled in five vertical stills of 10-tons capacity each. One still is provided with a mechanical agitator, vapor vent to the atmosphere, and a manhole on top. This still is used for making paints and mixtures as well as for tar distillation. The maximum capacity of this tar-distillation plant is 100 tons per week. Approximately 2 days are required for the dehydration and distillation of each batch of tar.

The primary products of the distillation are as follows:

Water: Average about 5 percent, but recent shipments have contained as much as 30 percent.

Heavy benzol: About 2 percent. It is used with additional heavy benzol purchased from others to make paints.

Middle oil, 16-18 percent) The middle oil and heavy oil are combined
Heavy oil, 10-12 percent) with pitch to make saturating tar for the roofing paper.

Briquette pitch (55°-60° K & S), 60-65 percent: Used for making paints, prepared tars, saturating tars, etc.

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The names of the more important coal-tar products of this firm and their approximate compositions are as follows:

Beton-Lubrose-Schwarz:

60 percent pitch (55°-62° K & S) + 40 percent heavy benzol. Used for the protection of concrete structures against industrial waters, moist earth, etc. Applied cold.

Heiss-Lubrose, Schwarz & Rotbraun:

95 percent pitch (55° K & S) and 5 percent asbestine. For hot application to concrete structures, foundations, walls, etc.

Carbolineum, Nussbraun:

A mixture of medium and heavy coal-tar oils. For brush or spray application to wood for protection against fungus.

Joserit-Carbolineum, Farbig:

An emulsion containing water, pigment, drying oils, and a small amount of carbolineum. For combined painting and fungus protection of wood.

Steinkohlenteer, Präpariert, Strassenteer:

Partly distilled tars or combination of pitch, heavy oil, and middle oil. Used for the impregnation of roofing felt, construction and maintenance of roads, etc.

Klebemasse:

Pitch cement and coating for use with the roofing paper.

Some of the products made from petroleum bitumen (asphalt) are:

Beton-Lubrose 714 b:

55 percent bitumen (70°-80°C. K & S) + 45 percent benzine. Used for drinking-water containers, reservoirs, tanks, etc.

Lubroplast A 111, Schwarz:

#1, 55 percent bitumen (45°-50° K & S) + 35 percent benzine + 10 percent asbestine.
#2, 55 percent bitumen (45°-50° K & S) + 45 percent benzine.

Jeserit-Bitumen-Emulsion D.R.P. 399557 Schwarz:

52 percent bitumen (45° K & S) + emulsifier.*

Note: (*) Rosin + NaOH before the war, bentonite during the war.

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Aluminium-Bitumen-Lubrose, Silbergrau:

80 percent Beton-Lubrose 714 b + 20 percent aluminium powder.

Silofarbe 1089, Schwarz:

50 percent bitumen, 90°-110° K & S + 50 percent benzine.

In addition to the above bituminous materials, this company made a phenolic resin paste called "copalit" for use on gaskets for pipes and machinery, and also a wide variety of colored paints in which, before the war, wood rosin, wood oils, and linseed oil were used. During the war these were not obtainable, and Albertol synthetic resins were used.

One of the principal products of the plant was roofing felt. It was saturated with tar, coated with filled pitch, and covered with sand or granules.

Conclusion

From the standpoint of tar distillation, this company offers little that is new or different from usual practice in Germany. Apparently little investigational work has been done and no effort has been made to produce improved coal-tar paints, coatings, road tars, pitches, etc. The roofing felt could not be evaluated because none was on hand and the plant had been destroyed.

Binne und Sohn, Hamburg, Germany

Tar-Distillation and Roofing-Felt Manufacture

The plant of Binne und Sohn, Pinneberg (near Hamburg), Mühlenstrasse 60, was visited on October 13 for the purpose, primarily, of seeing a plant where the manufacture of roofing felt is the principal line of business.

The persons interviewed were Dr. Hugo Binne and Wuerner Binne. They are co-owners of the company with their father. The business is quite old, having been founded by their grandfather, but the plant is modern with respect to its equipment as compared with other plants of this type that had been visited previously.

This plant was not bombed at any time, and all parts of it were in good operating condition. However, it was running at partial capacity for lack of sufficient raw materials. No stocks of finished materials were on hand, and there was only enough crude tar for 2 days' operation. The owners had been given no indication as to when more crude tar might be made available to them.

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The following information was obtained from the inspection of the plant and from discussions with the Messrs. Binne.

Tar dehydrated in the tar stills is mixed with pitch made in the same stills to produce a saturant for the roofing felt. For the coating material, to which sand is applied, pitch is mixed with 30 percent Kieselguhr or 40 to 45 percent calcium carbonate. The saturant and coating are produced as they are needed by the felt plant.

Byproducts of the tar distillations to make saturant and coating are light oil, sodium phenolate, and a mixture of neutral, medium, and heavy oil. The light oil is used for making black varnishes from coal-tar pitch or petroleum bitumen, the sodium phenolate is sold to Rütgerswerke for the recovery of tar acids, and the mixture of neutral oil and heavy oil is sold as carbolinum. During the war it was furnished to the Navy for use as fuel.

Before the war this plant made both asphalt and tar roofing felts, but during the war (as at present) only the tar product was made, because no asphalt was available for this purpose.

The equipment used for the various operations is as follows:

Tar Distillation

There are two 10-ton, vertical, steam-heated, flat-bottom stills equipped with mechanical agitators and vapor connections to water-cooled coil condensers, which discharge directly into small receivers. The stills are also connected to a vacuum pump, and vacuum of about one-half atmosphere is maintained during distillation. The agitators are used only when the stills are being used as mixers for making black varnishes from pitch and light oil or from bitumen and benzine.

Felt-Saturating Machines

Two felt-saturating machines are housed in separate buildings. Each consists of a saturating tank, a coating and sanding section, a looper, and a winder. Both machines were made by Otto Kinne at Halle/Saale. Each machine has a daily capacity (24 hours) of 30,000 square meters or about 90 tons of finished roofing.

Mixers for Coating Compound

One of the saturating plants has a horizontal, steam-heated mixer for making the coating compound. The other plant has two horizontal, coal-fired mixers for the same purpose. Previously, the steam-heated mixer was used for pitch-coating manufacture and the coal-fired mixers for asphalt-coating compound. The latter are now used for pitch-compound making, and the other plant is idle. Each mixer holds about 5 tons of finished coating material.

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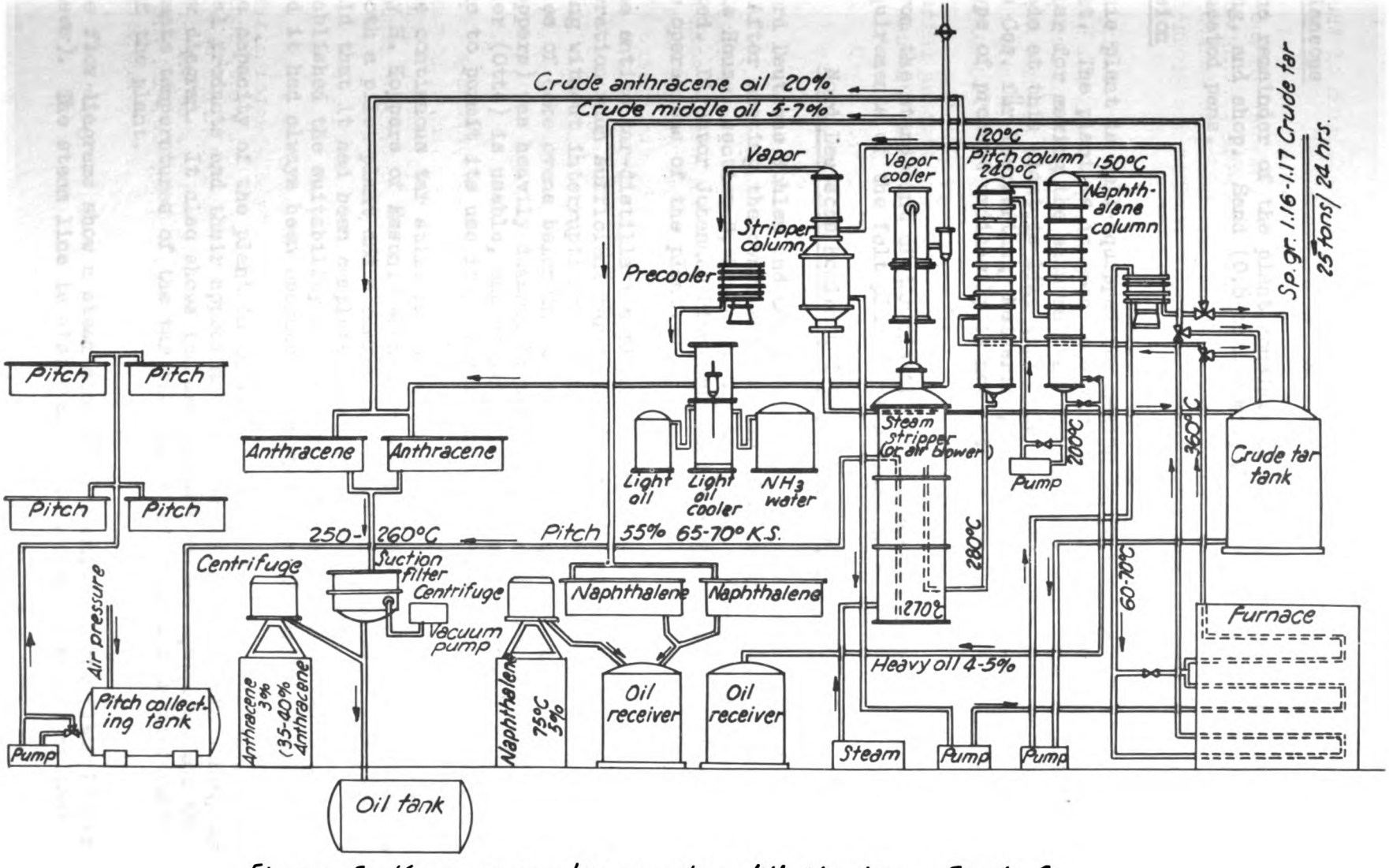

Figure 5.- Koppers continuous tar still - Hamburg - Freihafen.

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Miscellaneous

The remainder of the plant consists of office, boiler room, service building, and shop. Sand (0.6 to 1.0 mm.) for coating the felt is dried in steam-heated pans.

Conclusion

This plant is well-equipped for making tar-saturated and coated roofing felt. The plant is in good condition but lacks an adequate supply of crude tar for making the saturant and the coating. The type of roofing felt made at this plant is like that made at other tar plants in Germany such as Ges. für Teerwertung, Meiderich, and F. Raschig at Witten. The same type of product ordinarily is not made in the United States from coal tar.

From the standpoint of tar distillation (included only to take care of the requirements of the felt plant), this plant is unimportant.

Nord Deutsche Kohlen und Cokes Werke A.G., Hamburg

Nord Deutsche Kohlen und Cokes Werke A.G. was visited on October 13, 1945. After meeting the owner and manager, Erik Blümenfeld, at his office in Chile House, section B, Hamburg, the tar plant at Hamburg-Freihafen was inspected. Director Johannes Plückthun assisted Mr. Blümenfeld in explaining the operations of the plant.

The entire tar-distillation plant is completely undamaged and ready for operation when sufficient tar is supplied or produced to keep the still operating without interruptions. Formerly, the tar came from two adjoining batteries of coke ovens belonging to this same company. One of the batteries (Koppers) was heavily damaged by bombing and will have to be rebuilt. The other (Otto) is usable, and enough byproduct equipment will soon be operable to permit its use if permission to start operating again is granted.

The continuous tar still in use at this plant was the first of its kind built by H. Koppers of Essen. When it was installed in 1935 it was intended to be both a pilot plant and a commercial tar-distilling unit. Mr. Blümenfeld said that it had been completely satisfactory in both respects. It had established the suitability of the design for continuous tar distillation and it had always been operated with profit to the tar company.

The capacity of the plant is only 25 tons of crude tar per day. The principal products and their approximate yields are given on the accompanying flow diagram. It also shows the arrangement of the equipment and the approximate temperatures of the tar oil vapors and residues in different parts of the plant.

The flow diagrams show a steam connection (dampf) to the pitch blower (verblaser). The steam line is also connected to compressed air so that

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either steam or air may be used for blowing the pitch. When a maximum yield of anthracene oil is wanted, steam is used. When more pitch and less anthracene oil is wanted, air is used for blowing and, by polymerization, the pitch yield is increased. The pitch yield also is increased, at the expense of heavy oil, by returning a part of the latter as it comes from the bottom of the naphthalene column to the top of the pitch column. A part of this oil leaves the bottom of the column with the pitch and is converted into pitch in the verblaser by the air-blowing operation.

The light oil from this plant was combined with that from the coke ovens and worked up in the benzol-refining plant. The middle oil was crystallized in pans to recover naphthalene, which was centrifuged and marketed as crude naphthalene. The naphthalene drain oil was sold to other tar distillers, who extracted and refined the tar acids and combined the neutral oil with that from their own production. The heavy oil was used as wash oil by the coke plant, and the anthracene oil, after cooling and filtering to recover anthracene residue, was sold for hydrogenation or fuel-oil purposes. The anthracene residue recovered by crystallization and filtering was centrifuged before shipment to carbon-black makers. Its anthracene content was said by Director Plückthun to be 35 to 40 percent. The briquette pitch was used in the briquetting plant owned by this same company, where fines from its coal-washing operations were mixed with the pitch and briquetted.

The tube still contains two sets of tubes - one for dehydration and the other for distillation. All of the tubes are made of chrome steel. In the dehydrating section, according to Director Plückthun, the life of the tubes is about 4 to 5 years. In the distillation section it is 2 to 3 years.

The pipe still is heated with gas having a heating value of 4,500 kilogram calories per cubic meter. The gas consumption is about 55 cu.m. per ton of crude tar (containing about 3 to 5 percent water and having a specific gravity of 1.16 to 1.17).

Conclusion

With its combination of coal-washing plant, coke plant, tar plant, and briquette plant, this company's operations are well integrated. Incoming coal (by water) is washed. The coarse coal goes to the coke plant and the fines to the briquetting plant. Coking of the coarse coal produces coke, tar, etc. The tar is distilled to make oils and pitch, and the pitch is used to briquette the fine coal coming from the coal-washing plant.

The tar plant is arranged for efficient operation and is said by the owner to have been entirely satisfactory. No unusual features were observed other than the use of steam or air in the "verblaser" to vary the pitch and anthracene oil yields.

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Dr. F. Raschig G.m.b.H., Ludwigshafen am Rhein

The Ludwigshafen plant of Dr. F. Raschig G.m.b.H. was visited on December 6 and 7.

The persons interviewed were Dr. Kurt Raschig, Dr. Klaus Raschig, and Dr. Thönnessen. Dr. Kurt Raschig is the tar specialist, Dr. Klaus Raschig's speciality is synthetic resins, and Dr. Thönnessen is the specialist on organic derivatives.

This plant was bombed several times and was seriously damaged. For example, the synthetic phenol plant was 95 percent destroyed, the naphthalene-cooling house, 100 percent, warehouse, 80 percent, tanks, 80 percent, synthetic-resin plant, 60 percent, power house, 60 percent, hydrazine plant, 20 percent, pitch building, 100 percent, boiler house, 30 percent, etc. The building containing the four continuous tar stills was not destroyed, and all of them could be operated if enough tar were available.

Tar Distillation

The tar stills in use at this plant were developed by Dr. F. Raschig, the founder of the company. They have been described in the literature, so no detailed description of them will be given here. Briefly, however, each still consists of three shallow, cast-iron, vacuum pans mounted high enough above the ground so that all distillates and residues can be dropped to the first floor by barometric legs and withdrawn at atmospheric pressure. Each pan is about 5 feet wide, 9 feet long, and 8 inches deep. Baffles within the pans cause the tar to travel back and forth while passing from one end to the other. A pipe coil lying in the bottom between the baffles furnishes the heat for dehydration and distillation. The first two pans are heated by steam at 12 atmospheres pressure, and the third pan is heated by hot water under high pressure in a coil heated by coke. The total amount of steam used in the first two pans is about 150 kilograms per ton (metric) of tar. The third pan requires about 15 kilograms of coke per ton of tar distilled for heating the high-pressure water. The first pan operates at atmospheric pressure and is not provided with a fractionating column. In it the water and light oils are removed from the tar. The temperature leaving the still is 160°C. The second and third pans operate under 600 to 700 mm. vacuum and are equipped with columns packed with Raschig rings. Naphthalene oil comes from the bottom of the column on the second pan and carbolic oil from the top. The column receiving the vapors from the third pan discharges anthracene oil from the bottom and wash oil from the top. All of the condensates are discharged by barometric legs and seal pots to atmospheric receivers. Briquette pitch from the third still flows by gravity through a barometric leg, which also is a pitch-to-crude tar heat exchanger. From this heat exchanger the pitch flows to pans or split molds and the crude tar goes to the steam-heated, atmospheric, dehydrating still.

One complete distilling unit of the kind described above has a capacity of about 40 tons (metric) of crude tar per day, or 8,800 gallons. The fuel, power, and water requirements per ton of tar are as follows:

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Steam, 150 kilograms.
Coke, 15 kilograms.
Power, 4 kilowatts.
Cooling water, 4 cubic meters.

The Raschig plant has four units of the type described above for the distillation of coal tar. They are housed in one building, which was not damaged by bombs, although an adjoining building was completely destroyed. The men directly in charge of the tar plant are Dr. Hummel and Dr. Klamath.

Pitch Cooling

Two systems of pitch cooling are used at this plant - split molds and tilting pans. The split molds were filled in the usual manner on a covered platform. The roof was destroyed, but the platform is still usable.

The tilting-pan installation is comparatively new. In principle it is like the one used by Gesellschaft für Teerverwertung at Rauxel. Fifteen shallow, steel pans holding about 4 tons of pitch each are mounted beside a railroad track high enough above the ground so that the pitch will drop directly into a car when the pans are tilted. Each pan is tilted by a telescoping hydraulic jack, which moves the pan to an angle of 40 degrees with respect to the ground. Although the inside of the pan is coated with lime before it is filled, a small hydraulic pusher is provided at the center of each pan to start the movement of the pitch if it tends to stick. It is enough to loosen the pitch in one place to dislodge the entire charge.

Batch Tar Still

In addition to the four continuous stills described above, the Raschig plant has one 50-ton vertical tar still of the usual design for producing special grades of pitch or road tar without disturbing the continuous operation of the Raschig stills to briquette pitch.

Compound Mixing Equipment

Two horizontal coke-fired mixers with mechanical agitators are used for making special pitch compounds, such as paving fillers, paints, etc. The mixers are charged by hand-operated hoists, and they discharge by gravity into drums or other shipping containers.

Oil Redistillation

For redistilling contaminated tar acid-naphthalene oils received from others, or for redistilling naphthalene oil from which the tar acids have been extracted, two continuous Raschig units are used in a special installation provided for this purpose. Each unit consists of one steam-heated vacuum pan with packed column. When used for the redistillation of tar acid-free naphthalene oil, the latter is pumped to the vacuum pan at the rate of 1,000 liters per hour. The fraction from the top of the column,

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boiling below 200°C., goes to the light-oil plant, and the fraction from the bottom of the column (200°-240°C.) goes to the naphthalene pans. The residue from the still is returned to crude tar. The condensates and residue discharge through barometric legs to atmospheric receivers.

Naphthalene Recovery and Refining

The redistilled naphthalene oil, free of tar acids and tar bases, was cooled in 65 pans of 10 to 12 cu.m. capacity each. After draining, the naphthalene was centrifuged to make 70° + crude, or hot-pressed to make 79° crude. Crude naphthalene was refined in the usual manner, using two lead-lined washers (5 cu.m.) operated under 600 to 700 mm. vacuum and provided with 1.2 by 6.0 m. packed columns. The naphthalene-refining equipment was not damaged during the war, but the naphthalene pans and hot presses were destroyed.

Light-Oil Refining

The light oil-refining plant contains five 10-ton steam-heated stills and two 20-ton stills. All the stills are equipped with packed columns. Two large washers and one small one are on an elevated platform outside the light oil building.

Pyridine Recovery

Pyridine bases recovered from the tar acid-free naphthalene oil in the usual manner are distilled in two 5-ton steam-heated stills with packed columns, about 8 m. high and 65 cm. diameter. The grades of pyridine produced are pure pyridine, "test" pyridine (denaturing grade), and tar bases boiling above 160°C.

Tar-Acid Plant

Recovery of crude tar acids from freshly distilled carbolic oil is effected continuously by countercurrent flow of the oil and 8 to 10 percent caustic-soda solution through a vertical tower 1 by 16 m. filled with Raschig rings (35 mm.). The oil-feed rate is about 1,400 liters per hour, and caustic rate is about 2,000 to 2,400 liters per hour.

CO_2 from vertical batch lime kilns was used for springing crude tar acids. Batch causticizers and a batch filter were used for recausticizing the sodium carbonate. The $CaCO_3$ from the filter was used to neutralize acid wastes leaving the plant.

The crude tar acids are distilled continuously to make a rough separation of phenol and cresols. Two Raschig stills of the same size and type as those described above are used. The feed rate is 600 to 700 kilos per hour. The first still heats the tar acids to 160°C. and removes the water (without column). The second still, with packed column, removes overhead a mixture containing about 80 percent phenol and 20 percent cresol. The residue from the still consists of cresols and xylenols.

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The phenol fraction from above is redistilled in eight 5-ton stills using steam at 12 atm. for heating and 650 to 720 mm. vacuum. Columns are about 12 m. high by 65 cm. diameter. They are filled with 25 mm. Raschig rings.

For cresols, ten 5-ton stills are used with packed columns about 14 m. high, also one 50-ton still with a packed column about 14 m. high and 120 cm. diameter.

Residues from the cresol stills are redistilled in four 5-ton stills with 14 m. packed columns, and the residues from these stills are further distilled in one horizontal, 20-ton still equipped with a mechanical agitator. The still is heated by 12 atm. steam in a jacket, and a vacuum of about 600 mm. is maintained in the still. The distillate is cooled by a water-cooled condenser. The residue, just fluid enough to run our of a large gate valve in one end of the still, is loaded into steel drums and is finally returned to crude tar.

Symmetrical xylenol is made by cooling the fraction from the xylenol stills in steel cans about 30 by 8 inches lowered into a water bath. After crystallization of the xylenol, the can are warmed in a steam box, inverted over an inclined table, and the solid xylenol is crushed and centrifuged.

Chlorination of Tar Acids

Raschig chlorinates tar acids either with chlorine or sulfuryl chloride but prefers the latter, because it is more easily controlled and gives a more favorable yield of the monochlor compound. The chlorinating equipment consists of four vertical lead-lined tanks about 3 m. high and 1 m. diameter. Each tank has a working capacity of 1,700 kilos. A jacket is used either for cooling or heating. The lead-lined top is removable for inspection and is vented into tile pipe, which discharges fumes into a scrubber consisting of a tile-lined wooden tower filled with ceramic Raschig rings sprayed with water.

The chlorinating agent is introduced through a small pipe running to the bottom of the tank, and the contents, after chlorination, are discharged through the top of the tank by air pressure. There is no outlet on the rounded bottom of the tank.

The chlorinated mixture from the above reaction tanks is transferred by air to three 5-ton lead-lined stills with columns 60 cm. by 12 m., heated by steam and operated under vacuum.

Parachlor metacresol is made in the following manner: Meta-para cresol is chlorinated with sulfuryl chloride. The reaction mixture contains parachlor metacresol, paracresol, metacresol, and a small amount of dichlor metacresol. This mixture is fractionated to obtain cresols, parachlor metacresol, and pitch. The parachlor metacresol is dissolved in xylol and crystallized in three lead or enamel-lined half-round, jacketed, crystallizing pans. The crystallized product is then centrifuged to obtain pure parachlor metacresol.

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Dinitro-Orthocresol

This chemical is made by Raschig in the following manner: Orthocresol is sulfonated (with sulfuric acid in excess at 50° to 60°C.) in two horizontal sulfonators equipped with mechanical agitators. The sulfonated cresylic acid is transferred to small wooden tanks lined with acid-proof brick, where it is mixed with sodium nitrate and water, using a wooden paddle for stirring the mixture. A large amount of water is added for cooling and to crystallize the dinitro orthocresol. When the crystalline product is formed it is filtered by gravity through a canvas filter supported by a wooden grid. The wet dinitro orthocresol is shoveled into wooden containers and is shipped wet to avoid hazards involved in handling the dry product.

Dinitro paracresol is made in the same manner by the Raschig plant, starting with paracresol. The equipment used for making these products was not damaged.

Xylene Musk

Xylene musk is made by the Raschig company in the following manner: 22 kilograms of isobutylene (made by dehydrating isobutyl alcohol at 200°C. with alumina as catalyst) is reacted with 70 kilograms of metaxylene and a small amount of hydrochloric acid with aluminum chloride as catalyst at a temperature below 5°C. The impure isobutyl xylene made in this manner is washed with water to remove HCl and is fractionated to obtain forerunnings, 50 kilograms of isobutyl xylene and a residue. The isobutyl xylene is nitrated with a nitric acid-sulfuric acid mixture and cooled on ice to obtain 85 kilograms of crude musk. The latter is dissolved in benzine, distilled with direct steam to remove the benzene and impurities, and the musk, amounting to 85 kilograms, is recrystallized twice from pure alcohol. The final yield of pure musk is 68 kilograms.

Ketone Musk

For ketone musk, 80 kilograms of isobutyl xylene is reacted with 30 kilos of acetylchloride and 25 kilos of aluminum chloride for 1 to 2 hours at 20°C. The reaction mixture is cooled on ice, and the yield of ketone is 35 kilograms. The latter is nitrated with 98 percent nitric acid at -5°C., stirring for 1 hour, and the crude ketone musk is cooled on ice. It is washed with sodium carbonate, recrystallized twice from pure alcohol, and centrifuged. The yield of ketone musk is 26 kilograms.

Ambrette Musk

Pure metacresol is methylated with methyl sulfonic acid. The methyl compound is butylated with isobutylene, using aluminum chloride as catalyst, fractionated, nitrated, and crystallized from benzine to produce ambrette musk.

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Methylphenyl Ether (Anisole)

To make anisole, the Raschig plant reacts methyl sulfonic acid (made from methyl alcohol and sulfuric acid) with sodium phenolate. The reaction product is methylphenyl ether or anisole.

Paracresol methylether is made in the same way, starting with paracresol.

Chlorthymol

For chlorthymol, parachlor metacresol made as described above is fused and reacted with isopropyl chloride, using $FeCl_3$ as catalyst. After several hours, the reaction product is washed with water to dissolve the $FeCl_3$. It is then fractionated in vacuum to obtain forerunnings, consisting of parachlor metacresol, chlorthymol and a residue.

Xylene musk, ketone musk, ambrette musk, phenyl ethyl ether, paracresol methylether and chlorthymol were made only in small quantities by Raschig before the war and production was negligible during the war. The equipment was all small and of pilot plant type but well constructed and arranged for convenient and efficient operation. Most of this equipment was destroyed or badly damaged by bombs.

Hydrazine

Dr. F. Raschig G.m.b.H. claims to have originated the commercial manufacture of hydrazine in Germany and has made this product for many years. The hydrazine plant was operated at full capacity until almost the end of the war when it was damaged to some extent. The process consists in reacting NaOCl (made from chlorine and caustic soda) with ammonium hydroxide to obtain the hydrazine and sodium chloride.

Synthetic Phenol

The Raschig process for the catalytic synthesis of phenol at atmospheric pressure, using hydrogen chloride and atmospheric oxygen to form chlorobenzene, which, on hydrolysis with steam, gave hydrogen chloride for reuse and phenol, was developed at this location. A fairly large plant for carrying out the process was operated for several years. However, operation was stopped early in the war for lack of benzene, and later the plant was 95 percent destroyed by bombing.

Synthetic Resins

An extensive line of phenolic resins was made, including cast resins, resins for cementing, for plastic masses, for binding paper (hard-board resin), for paper manufacture, for grinding wheels, and water-soluble plywood glues. Novolak also is produced, and this company claims to be the only producer of Novolak using an alkaline catalyst. A horizontal, steel reaction kettle with mechanical agitator is used for Novolak production.

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The temperature used is 160°C. This information was reported to a resin specialist, who will investigate the Raschig resin plant more thoroughly and obtain further information about the Novolak production.

The resin plant was partly destroyed by bombing, but the Novolak department was in operation when visited. Some of the remaining resin kettles were being used for making disinfectants.

Disinfectants

Previously, Raschig furnished chlorinated cresols and xylenols to disinfectant manufacturers, who made high coefficient disinfectants. At present, the chlorinated tar acids cannot be furnished to the manufacturers, who are in another zone of occupation, and the products from those manufactrers are not available in the French zone, so the Raschig company is making large quantities of disinfectants themselves. Tin cans and plastic bottles previously intended for other purposes are being used for the distribution of these disinfectants.

Dr. F. Raschig, G.m.b.H., Langendreer

The plant of Dr. F. Raschig G.m.b.H. at Zweigniederlassung, Langendreer (near Bochum), was visited on September 19, 1945. The manager, Dr. Heuck, was interrogated, and the plant was inspected with him.

This plant is quite old and relatively unimportant, both from the standpoint of production and improved manufacturing procedures. During the period 1941-1943, the amount of crude tar distilled was about 38,000 tons per year.

Crude tar is dehydrated continuously by using the crude tar to partly condense the vapors from the tar stills. The dehydrated tar is then distilled in six vertical, coal-fired, 20-ton tar stills under 600 mm. vacuum.

The primary products are light oil, 0.5 percent; medium oil, 4.0 percent; heavy oil, 10 to 12 percent; anthracene oil, 12 to 15 percent; briquette pitch, 65 percent; or, when hard pitch is made, hard-pitch distillate, 10 percent, and hard pitch, 55 percent.

The medium oil is redistilled under 600 mm. vacuum in two 30-ton vertical batch stills with 1 by 12 m. packed columns to obtain carbolic oil and a residue that is added to heavy oil. The carbolic oil is crystallized in pans and centrifuged to obtain crude naphthalene and carbolic oil (1.2 percent). The latter is shipped to the Raschig plant at Ludwigshafen.

The heavy oil is redistilled in three 20-ton vertical batch stills with 1 by 6 m. packed columns to produce heavy carbolic oil, benzol wash oil (to coke plants), and residue (used in pitch compounds). The heavy carbolic oil is crystallized and centrifuged to obtain crude naphthalene (total 6 percent) and crystal-free, heavy carbolic oil. The latter is extracted with

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caustic, and the cresylate is shipped to Raschig at Ludwigshafen. The extracted oil is mixed with anthracene oil and sold for fuel purposes.

The anthracene oil is cooled in a vertical crystallizer with agitator and centrifuged to obtain anthracene residue and anthracene oil. The former went to carbon-black plants and the latter to fuel oil.

The briquette pitch is cooled in bays or split molds and is shipped to briquette plants.

The hard-pitch distillate is shipped to hydrogenation plants and the hard pitch to pitch-coke plants.

Some of the coatings and compounds made at this plant were produced as follows:

Felt saturant:	Distilled tar, about 20° K.S. for summer and 10° K.S. for winter.
Felt coating:	80 percent distilled tar, 25° K.S., and 20 percent stone-dust filler.
Roofing pitch:	(Klebemasse) soft pitch, +0°-+5°C.
Clay tube coating mass:	60° K.S. pitch, 50-52 percent, and 48-50 percent limestone-dust filler.
Black paint for iron:	60 percent 60° pitch and +0 percent heavy benzol.
Colored carbolineum, 91 percent:	(Anthracene oil, 60 percent, and water, 40 percent) sulfite lye, 3 percent and colored pigment, 6 percent.

Teerdestillation Stuttgart G.m.b.H.

Teerdestillation Stuttgart G.m.b.H., Stuttgart-Geisburg im Kienle No. 16, was visited on November 29. The tar plant is on the same property as the gas plant and works up the tar that is produced there. The amount of crude tar processed in 1943 was about 15,000 tons.

The manager of the plant is Mr. Hall, but he was not there at the time of this visit. The only persons interviewed were Miss Stenipfle, acting chemist, and two of the plant foremen. Miss Stenipfle said that the two men who had been responsible for running the tar plant and laboratory had been removed because they had been members of the Nazi party, and she was not well-acquainted with the operations or control of the plant.

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The tar plant was not running because of lack of tar. The gas plant had been severely bombed, and the coke ovens were damaged to such an extent that only a few were back in operation when the tar plant was visited. The production of tar at that time was only 3 to 4 tons per week.

The equipment of the plant consists of three stills, three mixing tanks for road tars, one tar acid-extracting tank, and a pitch-cooling shed. One of the tar stills is of the continuous Koppers type. A gas-heated pipe still and column arrangement similar to that of Nord Deutsche Kohlen und Cokes Werke at Hamburg is employed. This unit runs to briquette pitch, and its capacity is about 18 tons of crude tar per day. The other two units make only road tars. Their capacity is about 18 tons and 25 tons of crude tar per day, respectively. Each of these distilling units consists of a steam-heated still with a vapor to tar heat exchanger on top, which partly cools the vapors leaving the still and at the same time dehydrates the crude tar entering the still. In each type of installation the light oil from the dehydration of the tar is recovered separately, and the tar acid and naphthalene oils are combined. Wash oil and anthracene oil fractions also are recovered from the pipe still unit running to briquette pitch, but the other stills do not make these fractions because they run only to road tars.

The mixing tanks are equipped with mechanical agitators. They were used for making road tars containing pitch and heavy oil or for tar-bitumen road mixtures.

Tar acids are extracted from the tar-acid naphthalene oil in a vertical tank equipped with an agitator. Pitch is pumped to a loading station where, by means of troughs, it is run into split molds.

Conclusion

From the standpoint of improved processes or equipment, this plant is unimportant.

Ebano Asphalt-Werke Aktiengesellschaft, Hamburg

The plant of Ebano Asphalt-Werke Aktiengesellschaft at Hamburg-Harbourg, Moorbourgerstrasse 16, was visited on October 12 with Dr. Walter Becker and Hans Petersen.

The purpose of this visit was to obtain information about a special road-binding material (Spezialbindemittel) which this company was said to have made during the war from coal-tar pitch and tar oils.

The persons interviewed at this target were Dr. Walter Becker, Hans Petersen, and Joseph Schmid. The following information was obtained: Before the war there were large stocks of briquette pitch in Germany, and Ebano and others considered the possibility of using the briquette pitch with or in place of petroleum bitumen. Laboratory tests were made with 50/50 mixtures

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of briquette pitch and bitumen from Mexican petroleum, but the results were not satisfactory. When supplies of bitumen for road purposes became less available and the need for substitute materials increased, Ebano found that a distillate obtained by distilling pitch-coke distillate to coke with superheated steam and vacuum could be mixed 50/50 with bitumen, and, after blowing with air, this mixture appeared to be stable. The pitch coke distillate for this purpose came from the pitch-coke plant at Castrop-Rauxel.

Preparation of a road material in the manner described above was considered, but because of the work involved and the increasing shortage of bitumen, the General Inspector ordered that road binders should be made containing only coal-tar materials. According to Dr. Becker, the tar-distilling companies were asked first to make road binders using pitch and the pitch-coke distillate, but they did not care to take the responsibility for such mixtures, so the request was referred to Ebano. They accepted the request reluctantly, because prior to that time they had made only asphaltic road materials. Also, they considered it to be uneconomical to move so much raw material to Hamburg from the Ruhr. However, they proceeded to make a mixture of briquette pitch, pitch-coke oil, hard-pitch distillate, and neutral coal-tar oil (carbolic oil from which the tar acids had been extracted). The hard-pitch distillate was the oil obtained at the tar plants in distilling from briquette pitch (75°C., K & S) to hard pitch (150°C., K & S). The neutral oil was used in this mixture during the earlier part of the war as a solubilizing agent. Later it became unavailable, and anthracene oil was used in its place.

Dr. Hoepfner (an authority on bituminous road materials) expressed the opinion that this special binder was an ideal road material. At first, the road engineers did not like it, because it had to be heated to higher temperature (140°C.) than the grades of tar which they usually employed (110°C.). Later, according to Dr. Becker, they liked to use the new binder, although its viscosity was further increased and higher application temperature was needed owing to a lack of suitable fluxing oils.

In 1942, large supplies of Roumanian bitumen became available for use in the road materials, so the General Inspector ordered that this Roumanian bitumen be added to the "Spezialbindemittel." In addition to using available raw material, it was believed that it might make the road tar less "grainy" or crystalline and further increase its binding capacity. Dr. Becker is of the opinion that there may have been some improvement along these lines. The proportion of bitumen used in the mixture was 20 percent. The Roumanian bitumen had a penetration of 100 to 200 at 25° (100 grams, 5 seconds), R & B softening point 40° to 50°C., and contained 2 to 3 percent paraffins.

Still later in the war, some low-temperature tar from bituminous coal became available from Upper Silesia, so 4 to 5 percent of this tar was added to the mixture in making the special binder.

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The equipment used for making this product consisted only of tanks and a pump. Two tanks of 7,000 tons capacity each, equipped with air-agitating coils, were connected through a pump. After one of the tanks had been filled with pitch or a pitch-bitumen mixture and the other with the mixture of oils or oils and tar, the contents of the two tanks were mixed together by means of the pump and air agitation. The tanks and pump used for this purpose were totally destroyed by the air raids. More detailed information about this product is contained in a statement "Herstellung und Verwendung des Spezialbindemittel der Ebano Asphalt-Werke Aktiengesellschaft" furnished by Dr. Becker. Dr. Becker also furnished copies of the following documents:

Four laboratory analysis reports dated August 4, 1941, March 31, 1942, November 8, 1943, and February 7, 1944, covering "Spezialbindemittel."

Herstellung von Spezialbindemittel.

Spezialbindemittel-Erzeugung in den Jahren 1940, 1941, 1942, 1943, and 1944.

Emulsion Paint

During 1943 and 1944, Ebano made an emulsion containing the following materials:

35 percent "Spezialbindemittel" without bitumen.
10-15 percent of a mixture containing one-third clay similar to bentonite and two-thirds common clay.
50-55 percent water.

Three horizontal mechanical mixers mounted above storage tanks were used for making this emulsion. The clay was mixed first with a small amount of water to make a stiff paste. A small amount of caustic soda (0.5 to 0.8 percent, depending upon the alkalinity of the clay) was added next, and also the "Spezialbindemittel." The remainder of the water was then added and, after mixing thoroughly, the emulsion was dropped into the storage tank.

Camouflage paint for use on buildings, airport runways, roads, etc., was made from the emulsion by mixing it with mineral colors and water. The proportions were approximately 40 percent emulsion, 20 to 30 percent mineral pigment, and 30 to 40 percent water. Some of the pigments used were iron oxide, yellow ochre, carbon black, and a green pigment made by coating finely divided barium sulfate or calcium carbonate with a green aniline dye supplied by I. G. Farbenindustrie.

According to Dr. Becker, for paints of this kind it is necessary that the emulsified particles be fairly large, but not too large, 30 to 40 microns is preferred. Such an emulsion, even though prepared entirely from coal-tar materials, does not mask the color of the pigment like a similar emulsion having particle size of 3 to 5 microns.

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The quantity of camouflage paint of this kind that was made in 1943 and 1944, except in the winter months, average about 50 tons per day.

Plant Inspection

Following the discussion reported above, the plant was visited. Virtually all tanks were destroyed by air raids, and also almost all of the buildings and equipment except the three continuous petroleum stills. They were damaged somewhat but can be repaired without much difficulty. The laboratory building was damaged but now is used largely as an office building in place of the regular office building, which was totally destroyed. This plant was undamaged during the first three quarters of the war but was almost completely destroyed during the last quarter.

Emschergenossenschaft, Essen

Emschergenossenschaft, Essen, Germany, Kronprinzen Str. 24, was visited on November 23, 1945. The persons interviewed were Regierungsbaumeister Wiegmann and Dr. Schreiber.

"The Emschergenossenschaft was founded as a cooperative society legalized in 1904 by special law for the purpose of draining the entire Emscher district. This was done by regulating the natural and artificial drainage ditches, by building pumping stations, and by purifying the sewage. All towns and rural districts of the Emscher area, which direct all or part of their sewage to the Emscher River or its tributaries, have to be members of this cooperation."

One of the activities of Emschergenossenschaft is the extraction of phenols from the waste ammonia liquors from byproduct coke plants in the Emscher Valley. Twenty-four dephenolyzing plants were installed to recover phenolates that are sold by Emschergenossenschaft to Gesellschaft für Teerverwertung and Rütgerswerke (60 percent to the former and 40 percent to the latter). The total quantity of phenol and cresols recovered from this source by the two tar-distilling companies is about 5,000 tons per year.

All of the dephenolyzing plants operate on the same principle, although no standard design has been used for their construction. They were built by Koppers, Still, Kogag, and Otto.

The ammonia liquor to be dephenolized is washed continuously with benzol to extract the phenols from the ammonia liquor, and the benzol is then washed continuously with caustic soda to extract the phenols from the benzol. The phenolate is shipped to the two tar-distilling companies mentioned above, and the benzol is returned for the extraction of more ammonia liquor.

Ammonia liquor containing, for example, 4.13 grams per liter, is reduced by this method to 0.33 gram per liter, and liquor containing 1.05 grams per liter is reduced to 0.09 gram per liter before they discharge into the Emscher River.

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Lothringen Bergbau Aktiengesellschaft, Bochum-Lothringen

The pitch-coke plant of Lothringen Bergbau Aktiengesellschaft is near the Bochum-Gerthe tar plant of Gesellschaft für Teerverwertung. It was visited on September 17.

The ovens at this plant were not damaged by the air attacks, but piping was about 50 percent destroyed. Also, some damage was done to the roof over the quenching and loading platform.

The plant consists of 65 horizontal, fire brick-lined, sole flue ovens 1.5 meters wide by 10 meters long. Because heating through the sole flues alone was insufficient to calcine the pitch coke to less than 1.0 percent volatile matter, some gas was burned within the ovens themselves during the calcining period.

The ovens were charged with 150°C. pitch in crushed or flake form through charging holes on the tops of the ovens. The quantity of pitch charged was 14 tons. The coke was pushed from the oven by a ram onto a quenching and loading platform level with the floors of the ovens. Quenching water was applied by hand. The quenched coke was loaded into cars directly from the quenching platform.

The pitch distillate, amounting to approximately 15 percent of the pitch charged into the ovens, was distilled, in a special installation for the purpose, to hard pitch. The latter was charged into the ovens with hard pitch from the tar plants. The distillation of the pitch distillate to hard pitch was carried out with oxygen-free combustion gas. A small amount of coke-oven gas was burned in stack gases from the ovens. Combustion of the coke-oven gas was supported by the oxygen in the stack gas. By this method the stack gas was made oxygen free and at the same time heated to about 600°C. The oxygen-free stack gas then bubbled through the pitch distillate, which previously had been topped under about 400 mm. vacuum to remove light distillate for sale as fuel oil. The heavy oil obtained by distilling the topped distillate to hard pitch was shipped to hydrogenation plants.

The capacity of this pitch-coke plant was about 200 tons of coke per day. It was operated at full capacity throughout the war.

Verkaufsvereinigung für Teererzeugnisse, Castrop-Rauxel

The pitch-coke plant at Castrop-Rauxel was visited on September 17 and again at a later date. The persons interviewed on the first visit were Dr. Hochrle, director of Gesellschaft für Teerverwertung, and Dr. Klamm, manager of the Castrop-Rauxel tar plant of the same concern. On the later visit, Mr. Zumbusch, superintendent of the pitch-coke plant, was interviewed.

The pitch-coke plant suffered considerable damage from bombing and artillery. One block of five ovens was destroyed by a bomb, and much of the piping and steel work was severely damaged.

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The plant consists of 40 Koppers silica-lined, regenerative byproduct ovens built in two batteries of 20 ovens each. Each battery consists of four blocks of ovens with five ovens in a block. The blocks are separated by pinion walls, so that any group or block of five ovens can be operated or shut down independently of the other blocks.

The first battery of 20 ovens was built in 1939. The second battery was built in 1943. It was placed in operation in November of that year.

The dimensions of the ovens are as follows: Over-all length, 11,800 mm.; length between doors, 11,000 mm.; height, 3,000 mm.; average width, 450 mm. The 20 ovens in a battery are connected through standpipes to a common collecting main. The two collecting mains from the two batteries are, in turn, connected to two electrical precipitators, two indirect coolers, two direct coolers, and two exhausters. Decanting tanks separate the flushing liquor from the pitch distillate condensed in the collecting main, electrical precipitators, and coolers.

For charging the ovens with liquid, high softening-point pitch (150°C.), there were six vertical gas-heated tanks equipped with internal pumps. Each pump was connected to pipe loops that extended over the oven blocks. The molten pitch in a charging tank was kept in constant circulation through a loop by means of the pump. A float mechanism indicated the level of the pitch in the charging tank at any given time. The charging tank was filled with liquid pitch from an insulated tank car which transported it to the pitch-coke plant from the tar plant.

To charge an oven, a valve on one of the pitch-circulating loops was opened, and pitch flowed into the oven through a pipe inserted through one of the charging-hole covers. The introduction of the pitch was so regulated that 9 tons entered the oven during a period of 3 hours. Also, the rate of introduction was decreased during the 3-hour period according to a fixed schedule. During the first part of the charging period, a mixture of superheated steam and coke-oven gas was introduced into the oven through another charging-hole cover to minimize the cracking of vapors within the oven.

After 21 to 22 hours, the coke was pushed from the oven into a quenching car. A minimum amount of quenching water was used by covering the car, after the application of the water, with a steel cover provided for that purpose. The quenched coke had a water content of about 1 percent. After quenching, the coke was dropped onto a wharf and loaded into cars for shipment to the electrode factories.

Following are the data collecting during a 13-day test conducted in 1940. The pitch charged during this period had a softening point of 155°C., ash content of 0.3 percent, and specific gravity of 1,293.

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	13 days	Per day	Percent
Pitch charged	3,159 tons	243 tons	100
Pitch coke made	2,208 tons	165.9 tons	69.9
Pitch distillate	650 tons	50 tons	20.6
Gas (heating val.):			
min. 3,362 K cal.	785.000 Nm^3	60.400 Nm^3	7.0
max. 3,874 K cal.	79 tons	6 tons	2.5
Loss			

The analyses of the products made during this test period were as follows:

Pitch coke

	Percent
Water	0.8
Ash	0.4
V.M.	0.4
Pure coke .	98.4

Pitch distillate

	Percent
Water ..	2.0
Ash	0.025
Coke ...	18.5
S.P. ...	40°C. K.S.
Sp. gr.	1.262

Gas

	Percent		
CO ..	0.9	H_2S	3.09/m^3
CnHn.	1.0	HCN	0.59/m^3
O_2 ..	0.5	NH_3	2.39/m^3
CO_2 ..	3.5	Naphthalene	23.09/m^3
H_2 ..	11.8	Benzene ...	13.09/m^3
CH_4 .	13.0	Sp. gr. ...	0.877
N_2 ..	3.3		

The amount of gas produced by the ovens during the 13-day test was not sufficient for the entire needs of the plant (heating the ovens, boiler house, pitch-charging tanks, and door prewarming), and 193,640 Nm^3 of coke-oven gas was used in addition. Its minimum heating value was 4,099 K cal. The mixture of heating gases having a minimum heating value of 3,380 and maximum of 3,985 K cal. was distributed in the following manner: Heating the battery, 36.5 percent; boiler house, 45.5 percent; pitch-feed tanks, 14.5 percent; and door prewarmer, 3.5 percent.

Following are the tonnages of pitch coke produced by this plant during the period December 1939 to January 1, 1945:

	Tons
December 1939	522
1940	53,518
1941	64,007
1942	69,375
1943 1/	76,776
1944	77,724
	341,922

1/ New battery started November.

Mannesmannröhrenwerke Königin Elisabeth Mine, Essen

At this target, on November 23, the plants for briquetting bituminous coal fines and for producing pure coal for electrode coke were inspected.

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The briquetting plant was operating at part capacity when visited. It was using a stock of washed coal that was on hand, but no more was being produced. The plant had not been seriously damaged and could operate at capacity if enough coal were available. The capacity of the plant is about 400 tons a day (one shift). Briquette pitch is crushed to about 3/4 inch by an edge runner with perforated bottom. It is then elevated to a bin above a crusher, crushed in a hammer mill to 0-2 mm., and added continuously to crushed, washed coal (0-10 mm.) in the proportion of 6 to 7 percent pitch to 93 to 94 percent coal. The mixture is heated indirectly in large, shallow pans with revolving agitators. The coal-pitch mixture enters the pan at the center and is moved outward by rabble arms. It is then conveyed by a screw conveyor to vertical mixers, where steam is added in small amount. The mixture then drops into presses, where 3-kilogram rectangular briquettes are made. The pressure is about 150 atmospheres. The briquettes are transported to cars by conveyors but are loaded into the cars by hand.

The plant for producing pure coal for electrode-coke production was not operating for lack of raw material. It had not been damaged by air attacks. The process used in this plant involve (1) removal of "Finefrau" seam coal containing about 12 percent ash from the Emil shaft (about 930 m. deep); (2) removal of shale on a picking belt; (3) crushing to below 8 mm. and removal of fine dust; (4) screening to remove coarse dust (1 mm.); (5) laminar separation followed by spraying to separate refuse, briquetting coal, and the product that is floated to obtain pure coal containing about 0.5 to 0.7 percent ash. The yield of pure coal is about 27 percent. Apparatus was being installed for the electrostatic cleaning of the 0-1 mm. dust from the first screening operation. The process was to include drying in a Büttner turbo dryer, sifting in a vibro filter to about 0.1 mm., electrostatic separation, and flotation. This part of the plant was not completed. The older part of the plant, described above, produced pure coal that was mixed with 30 percent coal-tar pitch and carbonized in byproduct ovens at the König Ludwig coke plant.

Heinrich Koppers G.m.b.H., Essen

This firm was interrogated on September 22, 1945, concerning tar plant, pitch coke, and low-temperature carbonization equipment built in Germany and elsewhere. At a later meeting, the dephenolizing of waste waters was discussed.

This firm has built numerous continuous tar-distilling plants in England and Europe. The original and smallest plant is owned and operated by Nord Deutsche Kohlen und Cokes Werke A.G., Hamburg. The largest plant is owned by Kommanditgesellschaft Julius Rütgers, Mährisch-Ostrau. The Heinrich Koppers firm now has an agreement with Gesellschaft für Teerverwertung that will permit it to build continuous tar stills of the G.f.T. type as well as its own.

The arrangement of the continuous tar still built by Koppers for Nord Deutsche Kohlen und Cokes Werke is illustrated by a flow diagram included

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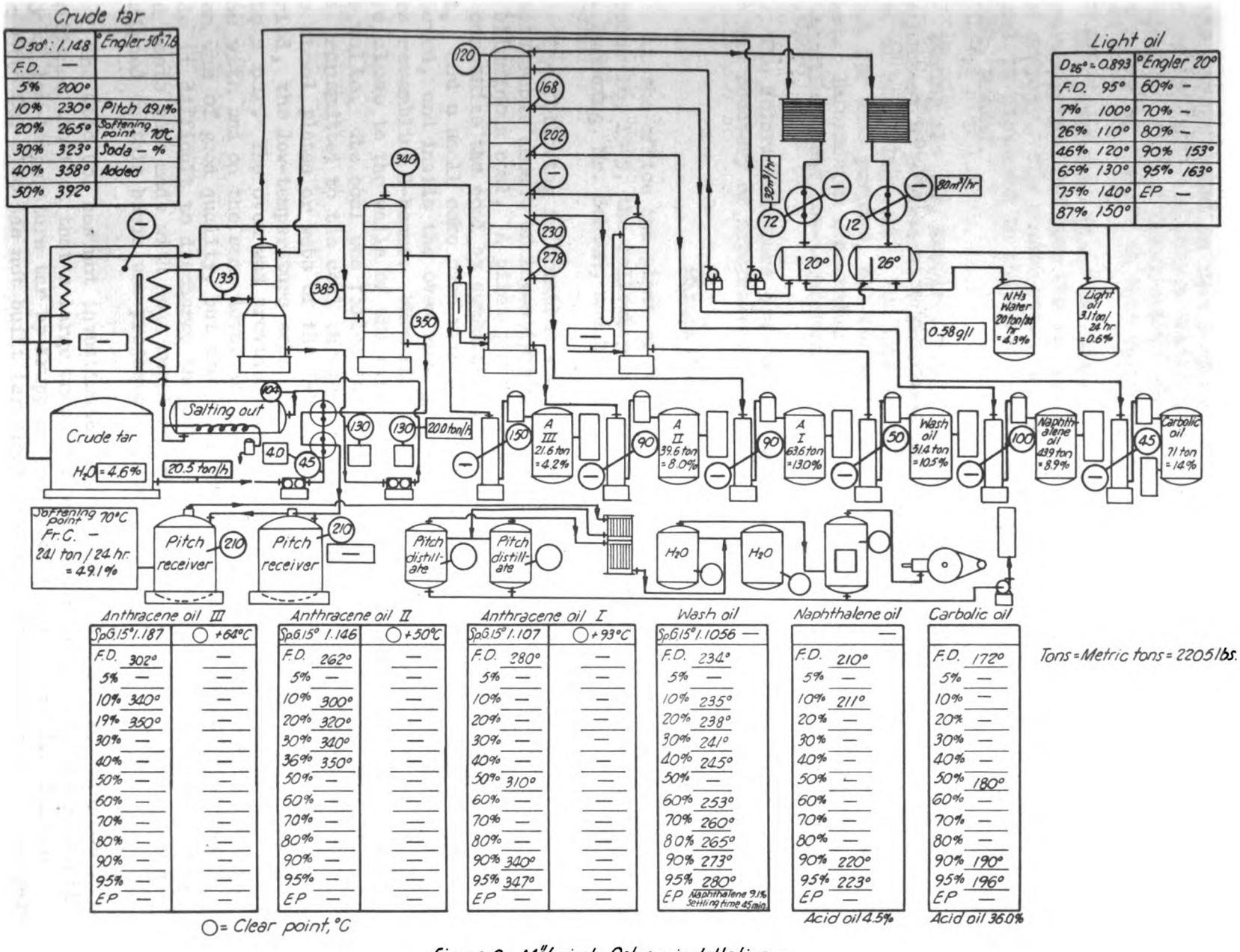

Figure 6.- Mährisch Ostrau installation.

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in the target report on that company. A diagram (fig. 6) showing the arrangement of the Mährisch Ostrau installation accompanies this target report. The continuous still of Gesellschaft für Teerverwertung is described in the report covering a visit to that company's plant at Duisburg-Meiderich.

The pitch coke plant at Castrop-Rauxel owned by Verkaufsvereinigung für Teererzeugnisse and operated by Gesellschaft für Teerverwertung was built by this company. The construction and operation of the pitch-coke plant at Rauxel is discussed in the target report on Verkaufsvereinigung für Teererzeugnisse, Rauxel.

During the war, Koppers, Otto, and Didier Werke collaborated in the development of a vertical refractory oven for the low-temperature carbonization of noncaking coal. The Koppers firm was authorized to construct a large plant for this purpose, but construction was not completed before the war ended. Information concerning this installation will be included with other information about low-temperature carbonization in a separate report.

The Heinrich Koppers organization has installed equipment at tar plants for the recovery of naphthalene, anthracene, and other coal-tar products.

Dr. C. Otto & Company, G.m.b.H.

At the office and plant of Dr. C. Otto & Co., Dalhausen, visited on October 16, 1945, the following persons were interviewed: Dr. Stuhlmann, Dr. Brensing, Mr. Schoen, and Mr. Boskamp.

The following information was obtained: This company has carried on a considerable amount of investigational work on low-temperature carbonization of bituminous coal. A pilot plant was built in which, at first, it was tried to carbonize the coal by sensible heat alone. This attempt was not successful, and a small coke oven with solvay-type horizontal heating flues was erected, and inside the oven were placed, in vertical position, steel members resembling I-beams. When they were in place, the flanges of the I-beams were close to the walls of the oven and the webs extended crosswise between the walls. The coal was filled into the spaces between the I-beams. Heat was transmitted to the coal through the side walls of the oven and also by the steel plates or webs of the I-beams. At the end of the carbonizing period, the low-temperature coke was dropped from the bottom of the oven into a car. The oven was provided with a regenerator, which was directly in line with and on the same level as the coke oven. The coke produced in this oven was of good quality, but the steel heating members warped badly and made it difficult to discharge the coke, so the steel members were removed and tests were made with heat supplied only through the side walls. A plant designed on this basis was projected but was not built.

The Otto Co. has not investigated low-temperature tar. Dr. Stuhlmann mentioned the fact that a large amount of work had been done on the miscibility of low-temperature tar by Krupp under Director Müller and Dr. Demann. The Otto Co. also has not built tar plants for the distillation of low-temperature tar.

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This company has built batch stills for companies that process high-temperature tar. In one installation in Italy, batch stills, with bell and tray columns mounted directly above the stills, were installed. In this case high vacuum was used during the distillation of the tar. In principle, this installation was the same as those which Rütgerswerke uses in its various plants.

During the war, this company shipped a continuous tar-distilling plant to the Japanese in Manchukuo. Difficulties were encountered in getting the various parts to the site near Mukden, but from telegrams received in 1943 the Otto Co. assumes that operations were started.

Recently, a plant similar to the one shipped to Manchukuo was projected for Switzerland, but work on the project was stopped and has not been resumed. A description of this plant, with a flow diagram attached and dated December 16, 1943, accompanies this report. The description also covers the Manchukuo plant in most respects. The tar still in the Manchukuo plant was provided with alloy steel tubes and cast-iron fractionating columns. The Swiss still was to be built with plain steel tubes and steel columns.

An oil-redistilling plant was built by the Otto Co. for Mathias Stinnes, and at other tar plants equipment for tar distillation, oil redistillation, naphthalene recovery and refining, and anthracene recovery has been installed.

Conclusion

The Dr. C. Otto Co. has been fairly active in the building of tar stills and tar-processing equipment. However, the most pretentious distillation units have been constructed for Japan and Italy. Installations built in Germany were all of the usual vertical or horizontal batch types without columns except on certain stills used for oil redistillation. A pitch-coke plant similar to the one at Lothringen was projected for Japan but was not built.

Description of a continuously working tar-distillation plant with tube furnace for the manufacture of light oil, washing oil, carbolic oil, anthracene oil, naphthalene oil, and briquette pitch. (See fig. 7.)

I. Dehydration

From the crude-tar tanks, in which the tar must be heated to a temperature that insures its being pumped easily, the tar is forced by one of the crude-tar pumps (1) through the heat exchanger, (2) in which the pitch is cooled while the crude tar is preheated to about 100°C.

The preheated crude tar now passes through the pipes of the dehydration section of the tube furnace, 3, where the temperature is raised to about 160°C. At this temperature, the crude tar is conveyed to the vapor liberator (dehydration column), 4, where ammonia liquor and light oil leave the top of the column and the dehydrated tar sinks to the bottom. The vapor mixture of ammonia liquor and light oil that leaves the top of the vapor liberator is

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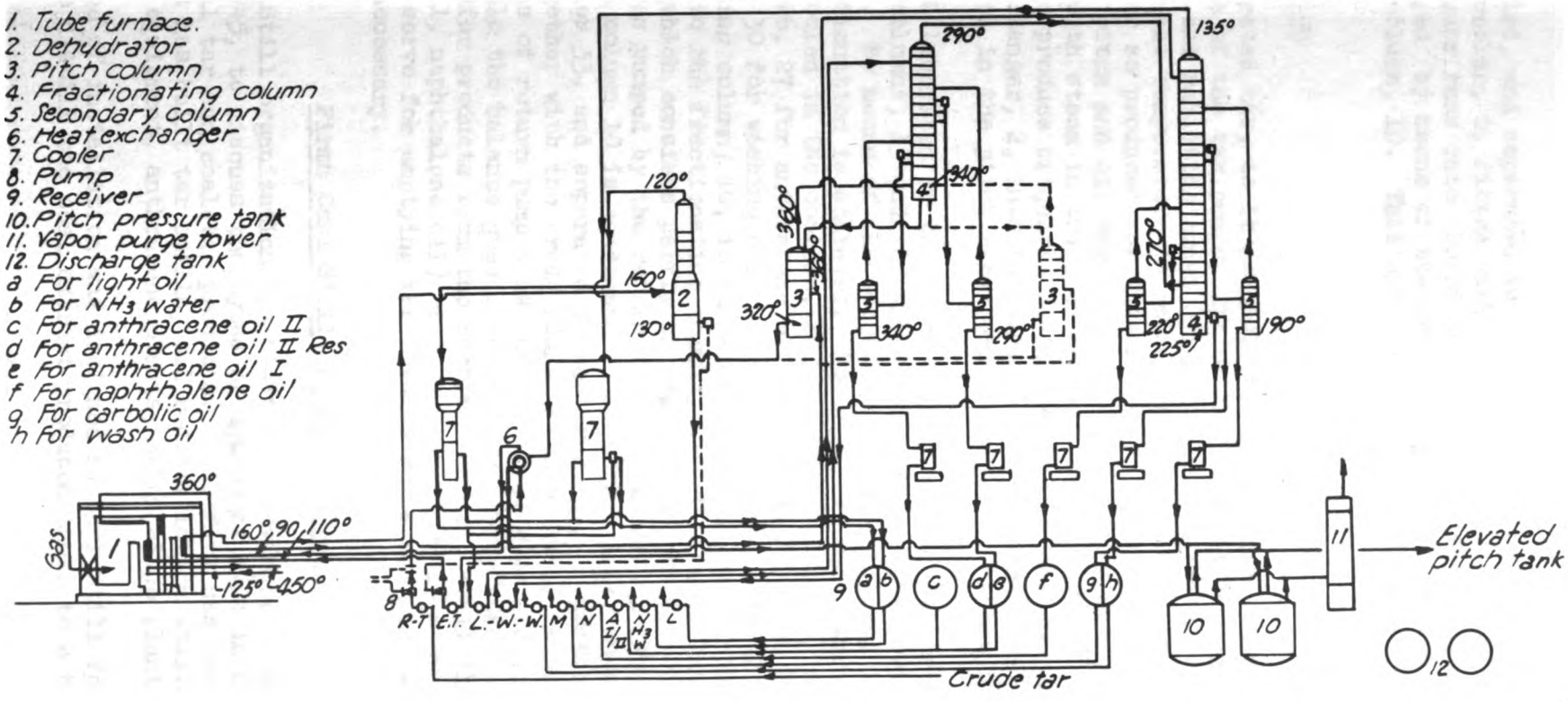

Figure 7.- C. Otto tar-distillation plant.

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condensed, cooled, and separated in the cast-iron plate condenser, 5, and in the light-oil cooler, 6, fitted with the decanter, 7. From the decanter, the ammonia liquor runs into the receiver, 8, whereas the crude light oil is normally conveyed by means of the return pump, 9, as reflux liquid to the fractionating column, 10. This operation finishes dehydration of the crude tar.

II. Distillation

The dehydrated tar, as it accumulates in the sump of the vapor liberator, 4, flows to one of the tar pumps, 11, which forces it in succession through the convection and radiating zones of the distilling section of the tube furnace, 3, while the temperature is raised to 350°-360°C. The mixture of liquor and vapor so produced now enters the pitch column 12, where it is separated into pitch and oil vapors. The pitch that sinks into the sump is treated there with steam in order to vaporize enough of the oil still present in the pitch to produce briquette pitch, which runs through the above-mentioned heat exchanger, 2, into the alternately operated pitch receiver, 13. Vapors liberated in the pitch receivers are absorbed in the fume tower, 14.

The vapors liberated in the pitch column pass in succession through two fractionating columns, 15 and 10, where they are separated into the individual fractions. By means of the accessory columns, 16, 17, 18, and 19, effective fractionation is achieved. The oils, as they flow out of the columns, are cooled in the coolers, 20, 21, 22, 23, and 24, and pass on to receivers 25, 26, 27 for anthracene oil, 28 for naphthalene oil, 29 for carbolic oil, and 30 for washing oil. A portion of the washing oil in the sump of the separating column, 10, is pumped by means of one of the pumps, 31, as reflux liquid to the fractionating column, 15. The vapor leaving column 10 is light oil, which consists partly of crude light oil from the vapor liberator, 4, that was pumped by the return pump, 9, as reflux liquid to column 10. The vapor from column 10 is condensed in cast-iron plate condenser 32, is cooled in cooler 33, and separated in separator 34. A portion of this top light oil, together with the crude light oil from vapor liberator 4, is pumped by means of return pump 9 as reflux liquid once more to fractionating column 10, while the balance goes into receiver 35 for production. Pumps 36 to 41 transfer products from the receivers to the available cooling pans (anthracene oil, naphthalene oil) or to the appropriate storage tanks, 42 and 43, which serve for emptying the furnace coils and the plant whenever this becomes necessary.

Firma Carl Still, Recklinghausen

The Carl Still organization was visited on November 9 and again on October 15, 1945, to discuss the types of equipment used in Germany for processing coal tar and coal-tar products. This firm has been active in this field and has built tar stills, oil redistillation stills with columns, pitch-cooling equipment, anthracene plants, naphthalene plants, etc.

A new type of installation was designed by Carl Still for the plant of Ruhröl A.G. to distill the coal extract produced there to a hard residue

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suitable for charging into coke ovens for pitch-coke production. The coal extract was pumped through a pipe heater, fired by gas, and released into either of two gas heated batch stills equipped with agitators arranged to clean the bottoms and side walls. At the outlet of the pipe heater an automatically operated valve maintained pressure on the extract while it was passing through the pipe heater. The operation of the two batch stills was so scheduled that the pipe heater could operate continuously and discharge alternately into the batch stills without interruption. The residue from the batch stills discharged onto an endless conveyor made of overlapping steel plates, which cooled the residue and discharged it into a car. The conveyor passed through water after discharging the pitch, to cool the plates before coating them with more pitch.

The largest installation of tar-processing equipment built during the war was constructed by this firm for use on brown-coal tar. The plant was built for the Aktiengesellschaft Sächsische Werke at Espenhain near Leipzig. The capacity of the plant was 200,000 tons of brown-coal tar per year.

It consisted of a continuous pipe-heater unit operating atmospherically, a vacuum, continuous unit to further distill the residue from the first unit, batch stills in which the residue from the second continuous unit was further distilled to hard pitch with inert gas, pitch-coke ovens into which the hard pitch was charged, and an Edeleanu extraction plant to remove paraffin from a part of the distillate that went to Diesel oil and fuel. The balance of the distillate went directly to fuel oil.

The Still organization built an oil redistilling unit for Carolinenglück consisting of four 50-ton horizontal stills connected to two offset, cast-iron, ball-and-tray columns.

Didier Werke A.G., Berlin

On October 31 Dr. Adolf Thau was interviewed at the office of Didier Werke A.G., Westfälische Str. 90, Berlin-Wilmersdorf. Matters pertaining to high-temperature and low-temperature coal tar were discussed. Subsequently, Dr. Thau submitted a brief statement, in writing concerning "Developments in the Technics of Tar Distillation in Europe."

The statement points out that vertical pot stills have been replaced to some extent in Europe by horizontal stills to obtain greater heat economy. Also, there has been a trend toward the use of continuous stills.

To use the stocks of briquette pitch that, at times, accumulated at tar plants, and at the same time to supply the need for pure coke for carbon electrodes, pitch-coke plants were erected in two locations.

Dr. Thau concludes that except for these two developments there has been little advancement in the field of coal tar-processing in Europe in recent years.

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A statement concerning the low-temperature carbonization of coal in Europe also was received from Dr. Thau. It is largely historical in nature. It will be covered separately with other documents on the same subject.

Schumachersche Fabrik, Bietigheim, Württemberg

On November 29 and 30, the factory of Schumachersche Fabrik at Bietigheim, near Stuttgart, was visited for the purpose of inspecting filters of the kind that were used by Ruhröl A.G. for filtering coal extracts. They were also used by various hydrogenation plants such as Hiedrierwerk Scholven, Gelsenkirchen-Buer; Rheinpreussen, Homberg-Mörs; Rheinischebraunkohle, Cologne; Wesseling near Cologne; and others for filtering middle oil produced by the hydrogenation of coal.

The filter rings for the pressure filters used by Ruhröl A.G. were supplied by Schumachersche Fabrik to Atlas-Werke A.G., Bremen, who made the filters. The use of these filters has been described briefly by this investigator in a separate report and in detail in the following CIOS reports:

Item 30, File XXXI-27, Coal-Extraction Plant of Ruhröl G.m.b.H., by H. H. Lowry and H. J. Rose, June 29 and 30, 1945.

Item 30, File XXX-104, Botrop-Walheim Hydrogenation Plant by C. Cockram, August 8, 1945.

The filter rings supplied to Atlas Werke by Schumachersche for this purpose are described by the latter company as "Braungelb #50 filter rings." Their dimensions are 125 mm. O.D., 81 mm. I.D., and 30 mm. high. When the factory was visited, 20,000 of these filter rings were on hand. Each of the Atlas filters at Botrop contained 32 candles, and each candle was made up of 60 filter rings, so that about 1,920 rings were required for each filter. The rings were replaced after 5,000 filtering cycles of 15 minutes each.

Schumachersche Fabrik furnished complete filters to the hydrogenation plants. Their No. 100 filter was supplied to those customers. Each filter contained one filter tube 1 meter long and 300 mm. O.D. made up of Braungelb #50 rings of 300 mm. diameter. The larger-diameter rings could be used for filtration of medium oil at hydrogenation plants, because the liquid to be filtered was much cleaner than the coal extract at Botrop, and lower filtering and cleaning pressures were employed. A single hydrogenation plant used as many as 40 filters at one time.

When the Schumacher factory was visited, it had on hand about 100 No. 42 filters. These filters hold one candle 420 mm. long and 125 mm. O.D. The candle is made by cementing individual rings together with a phenolic resin and then turning the tube to proper diameter and length. One No. 42 filter, complete with filter cylinder, brown yellow No. 50, and three reserve cylinders of the same kind were purchased for shipment to America for experimental purposes. The price of the complete filter was 195 RM, and each additional cylinder cost 12.20 R.M. Cost of case and packing was 12.0 R.M. In addition to the above, two "Carbo" No. 40 filter cylinders to fit the No. 42 filter

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were purchased and packed for shipment to America. These filters cost 15.70 R.M. each and packing cost 3.0 R.M. Alkaline liquids can be filtered with the Carbo filters, whereas the "Braungelb" filters must be used with neutral or acid liquids.

The numbers 40 and 50 refer to the grain sizes and porosities of the filter tubes. Porosities range from fine (No. 10) to coarse (No. 60). No. 50 has been found to be the most suitable porosity for medium oil from hydrogenation plants and for coal extracts of the kind produced at Botrop.

In using the No. 42 filter equipped with No. 50, "Braungelb," cylinder, the working pressure should be about 8 kg. per sq. cm., and the highest permissible pressure is 12 kilograms per sq. cm. When cleaning the filter with air, the pressure should be about 4 kilos per sq. cm.

Filter rings are manufactured entirely by hand at Bietigheim. A weighed amount of "Chamotte" is poured into a mold, subjected to high pressure with a hydraulic press, and the ring is dried atmospherically and then fired in gas-heated kilns.

The persons dealt with at this plant in purchasing the equipment were Albert vom Hofe, Helene Martin, and Gottlieb Xander. Army headquarters at Esslingen and the Military Government for Bietigheim were consulted before purchasing the equipment.

Aktiengesellschaft der Kohlenwertstoff-Verbände, Bochum

The offices of Aktiengesellschaft der Kohlenwertstoff-Verbände were visited on September 13, 1945, and several times thereafter. At present, the offices of this organization are at Waldring 71 (Schillerschule), in Bochum. The central office building previously occupied in Bochum was totally destroyed by air raids, and most of the records, stored in safes in the basement, also were destroyed.

Kurt Haver, director of Kohlenwertstoff-Verbände, with the permission of the Military Government, assisted materially in collecting information about the German coal-tar industry, arranged appointments for visits to some of the tar plants, and assigned Dr. Geiselbrecht, of his organization, to accompany the investigator to some of the targets as guide and to assist in the interrogations.

During the war, Mr. Haver's organization directed, for its associated members, the production and distribution of nitrogen, benzol and benzine, coal-tar products, coumarone resins, phenol, and methane. The individual groups brought together during the war to form the organization known as Kohlenwertstoff-Verbände and the products which they handled were as follows:

1. Deutsche Ammoniak-Verkaufs-Vereinigung (D.A.V.V.): Nitrogen obtained from synthetic plants, coke ovens, and gas works.

2. Benzin-Benzol Verband (B.V.): Benzol and benzine.

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3. Verkaufsvereinigung für Teererzeugnisse (V.F.T.): Coal-tar products.

4. Coumarone Verband (C.V.): Coumarone resins and residues from coke plants and tar plants.

5. Phenol Verband (P.V.): Phenol from tar plants and from synthetic phenol plants.

6. Treibgas Vereinigung (T.V.): Methane from gas wells for use as motor fuel. Another group of Kohlenwertstoff Verbände, "Gruppe Schwefelsauregemeinschaft" (S.G.), purchased all supplies of sulfuric acid for the coke ovens, synthetic-nitrogen plants, and metal-working factories belonging to its associated members.

Mr. Havor and Dr. Geiselbrecht furnished the investigator with product specifications and miscellaneous statistical information concerning German coal-tar products.

Phenol-Verband, Berlin

An attempt was made on October 21, 1945, to find Dr. Westermann, director of Phenol-Verband, at his office in Berlin, Gross Admiral von Kosfer-Ufer, 59. The building was badly damaged, but a secretary was found in the office who thought Dr. Westermann might come there the following day. A return visit by the writer could not be arranged, but subsequently Dr. Müller, president of Phenol-Verband, was found in the office of Rütgerswerke A.G., and he furnished the following information:

Phenol-Verband now has only two members in its organization in eastern Germany, namely, Rütgerswerke and I. G. Farbenindustrie at Leuna. All of the other former members are now in the Polish sector and are no longer German firms.

The I. G. plant at Leuna recovers tar acids from the water resulting from the distillation of brown coal and from oils produced by the hydrogenation of brown coal and brown-coal tar. Previously, the Leuna plant produced 24,000 tons of phenol per year.

Rütgerswerke had a synthetic-phenol plant at Niederau (now dismantled for shipment to Russia) and also extracted phenols and cresols from tar oils. Its production was about 2,500 tons of phenol and 4,000 tons of cresols per year. It now is about 240 tons of phenol and 480 tons of cresol per year.

The total output of phenol by the two companies was said by Director Müller to be about 60 to 100 tons per month at the present time.

Verkaufsvereinigung für Teererzeugnisse, Berlin-Wilmersdorf

The Berlin office of Verkaufsvereinigung für Teererzeugnisse, Berlin-Wilmersdorf, Hohenzollerndamm 42a, was visited on October 30, 1945, to obtain

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information about the production and sale of coal-tar products, particularly in the central and eastern parts of Germany.

The persons interviewed at this target were Director Weger and Director Weiss. Previously, this organization sold the products of all of the tar distillers in eastern Germany. At present, it has very little to sell, because tar production is only about 5 to 10 percent of normal, and because most of the tar plants that were under German control during the war are now destroyed or under Russian or Polish control or ownership. V.f.T. sells small quantities of pitches and oils from Erkner that the Russians do not want for their own use, but benzol, naphthalene, phenols, creosote, and other products are not made available for sale by V.f.T.

Following is the status of each of the tar plants of eastern Germany according to Weger and Weiss:

Rütgerswerke Erkner - in order and working at part capacity.
Rütgerswerke Breslau - completely destroyed (beyond repair).
Rütgerswerke Niederau - equipment about to be removed to Russia; nothing left but the buildings.
Rütgerswerke Teerag, Wien - no information; presumed that plant exists.
Borsig (Scala) Hindenburg - benzol and tar plant completely removed to Russia.
Odertal, Gleiwitz - Polish, but no news as to condition.
Oberhütten - plant is working, but for the Poles.
Kattowitz - Polish; no news as to condition.
Emmagrube, Kattowitz - Polish; no news. (only made pitch and oil).
Döbeln - German, but no news (said by Kahl to be in order).
Zwickau - German, but no news (said by Kahl to be in order).
Velten - German, but no news (said by Kahl to be in order).
Waldenburg - German (Polish, according to Kahl); no news.
Küstrin - Polish; completely destroyed by war.
Danzig - Polish; no news (said by Kahl to be in order).
Rütgerswerke Preussen - Russian; condition unknown.

According to Weger and Weiss, the future of the German tar industry will depend on (1) production of tar, (2) attitude of the Russians, (3) availability of materials for reconstruction.

I. G. Farbenindustrie, Ludwigshafen

At the Ludwigshafen plant of I. G. Farbenindustrie, visited on December 5 and 6, some of the wartime uses for coal-tar products at that plant were investigated. The persons interrogated were Dr. Snell, Dr. Fischer, Dr. Winmer, and Dr. Johannsen.

According to Dr. Snell, the principal uses for naphthalene at Ludwigshafen were for phthalic anhydride manufacture, about 550 tons per month; for beta naphthol, about 500 tons; and for tanning agents, detergents, and dyestuffs, about 150 tons per month. The latter, i.e. tanning agents, detergents, and dyestuffs, were made in greater quantities at Leverkusen than

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at Ludwigshafen, according to Dr. Snell. Naphthalene also was used at Ludwigshafen for the manufacture of naphtylamines.

Brief visits were paid to the plants in which phthalic anhydride, maleic acid, and koresin were made. These plants have been discussed in reports submitted by other investigators, so no attempt was made to cover them in detail.

In the phthalic anhydride plant, two units of 80 to 100 tons capacity each were operating and making about 150 tons of phthalic anhydride per month. It was expected that the capacity would be doubled in about 3 months, when two more units would be ready for operation. Originally there were seven units in this plant, but three were destroyed by air attacks. The two units that were operating had been started without difficulty, although the catalyst was approximately 6 years old. The construction and operation of this plant is discussed in CIOS Report Item 22, File XXVII-85, by Kern, Murray, and Sudhoff.

Maleic acid is made at Ludwigshafen from crotonaldehyde instead of benzene. It was explained that after 1933 "cheap benzol" from America was no longer available, and it became less expensive to use crotonaldehyde. It was stated, however, that if cheap benzol becomes available again it may replace the crotonaldehyde.

The process used in the maleic-acid plant also has been discussed in CIOS Report Item 22, File XXVII-85. The maleic-acid plant was not operating when visited. It was heavily damaged by air attacks and no repairs, either to the building or equipment, had been started.

Koresin was made by reacting isobutylene with phenol, using aluminium chloride as catalyst, to obtain para-tertiary-butyl phenol. The latter was then reacted with acetylene, using zinc naphthenate as catalyst, to obtain the koresin. This process has been described in CIOS Report Item 22, File XXIX-62, and the Quartermasters Technical Intelligence Report on "The German Plastics Industry." When visited, this plant was operating at part capacity.

Phthalic Anhydride and Alkyd Resin Manufacture by I. G. Farbenindustrie, Uerdingen, Germany

The plant of I. G. Farbenindustrie at Uerdingen, Germany, was visited on October 1, 1945, in order to obtain information concerning the manufacture of phthalic anhydride and the use of this chemical in making alkyd resins and phthalates.

The persons interviewed were Dr. Dilthey, assistant manager; Dipl. Ing. Kutsch, engineer in charge of the construction of the phthalic anhydride plant; Dr. Hamann, Director of Research; and Dr. Kinsky, superintendent of the alkyd resin plant.

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Dr. Dilthey stated that there are only three phthalic anhydride plants in Germany - I. G. at Oppau-Ludwigshafen, I. G. at Schkopau (near Leuna), and I. G. at Uerdingen. Construction of the Uerdingen plant was begun in 1942. When it was about 80 percent completed, construction was stopped because of the greater necessity for work along other lines. Construction was resumed a short time ago by permission of the Military Government. It is expected that the plant will start production shortly after the first of the year. The plant suffered no damage from air raids except broken windows.

The phthalic anhydride plant is housed in two buildings. One building is devoted entirely to receiving, storing, melting, and filtering of flake naphthalene made at tar plants in the Ruhr. The flake naphthalene, in paper bags, arrives in railway cars or trucks and is dumped into a pit, from which it is elevated by a crane to a storage platform. It then is fed by the crane into a hopper above a crusher. After passing through the crusher, the naphthalene is elevated to an overhead bin, from which, by means of a screw feeding mechanism, it is filled into an automatic weigh tank suspended from a trolley. The weigh tank discharges the naphthalene into three steam-heated melting kettles equipped with mechanical agitators. The molten naphthalene flows by gravity through three filters containing "zellwolle" to pumps, by which it is transferred to six other filters (three large and three small) in the top of the building. From there it drops into steam-heated storage tanks.

The second building contains, in one end, offices, service facilities, and plant laboratories. The next section of the same building contains eight steam-jacketed tanks, which feed the molten naphthalene to the vaporizers. There are two feed tanks for each vaporizer. On the floor below the feed tanks, and directly below them, are the instrument-control panels. On this same floor are the air-to-air heat exchangers, vaporizers, and converters. In a lower portion of the same building adjoining the portion that houses the offices and laboratories are the various air pumps required for feeding air to the converters and to the heat exchangers. There are eight blowers for each of these purposes (two blowers of each kind per converter).

Vaporizing of the naphthalene and conversion to phthalic anhydride are carried out as follows: Molten naphthalene from the overhead feed tank flows through a rotameter into the vaporizer, where it is vaporized by air preheated in the heat exchanger by hot air from the cooling section of the converter. The mixture of air and naphthalene vapors passes downward through the converter and to the condensing chambers. There are fourteen chambers for each converter. The first four are water-jacketed for cooling and the last ten are air-cooled. Each cooling chamber is equipped with a screw conveyor at the bottom, which discharges the phthalic anhydride into a Redler conveyer running below the 14 chambers and to the melting kettles.

The construction of the converters is as follows: 3,000 tubes of 25 mm. diameter are arranged concentrically between tube sheets, around a central pipe about 24 inches in diameter. The catalyst is placed inside the tubes, and a mixture of salts used for cooling the converter surrounds the tubes. During operation, a molten-salt bath is circulated through the central

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tube by means of an impellor in the top of the tube, which is driven by a motor mounted on top of the converter. Cooling of the salt is accomplished inside the central tube by air circulated upward and then downward through double-walled pipes, which are closed at the upper end (air rises in the outer pipe to the closed end at the top, then reverses and goes down through the inner pipe). The hot air from this cooling system next circulates through the tubular heat exchanger, where it preheats filtered air going to the steam-heated vaporizers, and from there goes to the converters. In the vaporizers the preheated air flows over the molten naphthalene and becomes saturated with naphthalene vapor.

The cooling chambers are made of ordinary steel. They are about 6/10 meters wide by 4 meters long and 4-5/10 meters high. A single partition inside causes the vapors to pass downward and then upward in passing through the chambers. From the last chamber, the air passes through a cyclone dust collector, exhaust fan, and stack to the atmosphere.

The "hay" from the cooling chambers goes, by means of the Redler conveyor, to the four steam-jacketed melting kettles equipped with agitators. Vapors from these melters are scrubbed by water in Lurgi mechanical scrubbers. From the melting kettles the molten crude phthalic flows by gravity to storage tanks (steam-jacketed). From the latter it flows to two refining stills heated by hot water under 68 atmospheres pressure (equipment made by Bamag-Meguin at Giessen). The refining stills are equipped with packed columns, water-cooled condensers, overhead receivers for separating vapors from condensate, and barometric legs which deliver the condensate to small jacketed tanks, which feed the flaking rolls. From the latter, the flaked phthalic anhydride goes to the storage bins.

Vapors from the refining still condensers pass to sublimate catchers, then through caustic-soda scrubbers, and to the vacuum pumps. Residues from the two large refining stills are transferred to two small hot water-heated stills for further distillation. The vapors from these stills are handled in the same manner as those from the large stills.

According to Dr. Dilthey, the naphthalene used in this plant will have a freezing point of 80°C. The catalyst will be of the vanadium pentoxide type. Enough catalyst is on hand to charge the converters at the start. This catalyst probably was made at Ludwigshafen.

According to Mr. Kutsch, it is expected that the catalyst will last at least 10 years and that the converters will not need to be opened in that time. Two of the converters intended for this plant did not arrive before shipments of materials to the plant were stopped. They probably are at the Ludwigshafen plant.

Other Products Derived from Coal

Following the visit to the phthalic anhydride plant with Mr. Kutsch, various products made by I. G. Farbenindustrie were discussed briefly with

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Dr. Dilthey and Dr. Hamann. Those products made from materials derived from coal were of especial interest.

Crude benzene is refined at this plant. From the pure benzene are made large quantities of aniline by nitration, followed by reduction with iron. Yellow and black iron oxides are produced as byproducts, which were said to be excellent pigments for use in the building industries.

Toluene is chlorinated to make benzyl chloride, benzal chloride and benzene trichloride. By the use of alkalies, benzyl alcohol, benzaldehyde, and benzoic acid are produced. Benzoic acid also is made from toluene, using sodium bichromate, by the Bozel-Maletra process. Parachlorbenzoic acid is produced for use in making saccharine.

Solvent naphtha from the crude benzol-refining plant is used in the resin plant.

Naphthalene will be used after the first of the year for making phthalic anhydride, as stated above. However, in the past large amounts of phthalic anhydride produced by I. G. at Ludwigshafen have been used at Uerdingen for making alkyd resins.

Alkyd Resins

Following the general discussion reported above, the alkyd resin plant was visited with its superintendent, Dr. Kinsky. It is all housed in one building on one floor and balcony. On the balcony are 11 stainless-steel resin kettles equipped with agitators. Seven of these kettles are of 4-1/2 tons capacity each and are heated by gas (the burner is located below the kettle in a jacket that extends up the sides almost to the top). These kettles have stainless-steel coils on the inside for cooling with water. The other four kettles are of 6-1/2 tons capacity and are heated by steam from the boiler house at 60 atmospheres pressure. These same coils are used for cooling (with water).

No vacuum is used on the kettles, but inert gas is bubbled through the reaction mixture each time a batch of resin is made. The inert gas is also used for blowing the finished resins to the zinc-coated drums in which they are shipped. The inert gas used for this purpose is usually nitrogen made by the Linde process, but carbon dioxide also is used at times.

The principal raw materials used in this plant are phthalic anhydride, glycerol, hexanetriol, pentaerythritol, 3-methalol propane, 3-methalol ethane, castor oil, linseed oil, tung oil, and vorlauf fettsäure (a product derived from Fischer-Tropsch paraffin).

According to Dr. Kinsky, alkydal T, suitable for air-drying and baking purposes, comprised at least 75 percent of the total output of this plant. This product contains the following: 55 percent linseed oil, 18 percent glycerol, and 35 percent phthalic anhydride. The linseed oil and glycerol

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are mixed together and heated at 260°C. for 5 to 6 hours, or until the mixture is soluble in alcohol. The mixture is cooled to 150°C., and the phthalic anhydride is added. The temperature is then raised to 240°-250°C., and the product is sampled and tested for acid value and viscosity. When proper values are reached, the product is cooled to 150°C. and is filled into zinc-coated drums. Alkydal T is made in three consistencies - TN, TM, and TH. Solutions of these grades of alkydal T are made in benzene, using 25 to 40 percent benzene. (The T comprises 60 to 75 percent of the final solution.) The solutions are made in mixing kettles on the ground floor, below and in front of the resin kettles that stand on the balcony. The solution kettles are provided with mechanised stirrers and means for heating and cooling.

According to Dr. Kinsky, comparatively small amounts of nondrying resins are made. They contain castor oil or vorlauf fettsäure. A slow drying resin called alkydal ST also contains vorlauf fettsäure.

In addition to the alkyd resins, this plant makes large amounts of a plasticizer called Eleol. It is a plasticizer for nitrocellulose and polyvinyl chloride. It contains 3 mols of vorlauf fettsäure (3,200 kilos) and 1 mol of hexanetriol (1,300 kilos). These ingredients are heated together in a resin kettle with inert gas and mechanical agitation to 150°-190°C. Water distills off during this period. The reaction product is then cooled to 50°-60°C. and washed with sodium carbonate solution to remove excess vorlauf fettsäure, then centrifuges to separate the raw Eleol and soap solution, dried by heating in a kettle to 100°C. while agitating with inert gas, bleached with tonsil (activated carbon), filtered through a plate and frame filter press, and transferred to storage tanks or drums.

The alkyd resin plant was undamaged by the war and is able to operate at full capacity if sufficient raw materials are available.

I. G. Farbenindustrie, Schkopau

The primary purpose of a visit to this target on January 5 was to inspect a Phenosolvan installation for the recovery of phenols from waste waters.

Information to the effect that there was such an installation at Schkopau proved to be incorrect. However, Dr. Alfred Dierichs, who had worked with the process at Leverkusen, furnished a copy of an article written by himself, and published in Chemiker Zeitung, 1942, volume 66, Nos. 27/28, page 288. This document has been microfilmed with documents on dephenolization of waste waters collected from other sources.

A list of the products manufactured at Schkopau was requested in order to determine to what extent products derived from coal tar were employed as raw materials at that plant. The accompanying list of products was obtained. It gives the capacity of the plant for each chemical in May 1945 and also as it would be after damage caused by air attacks had been repaired.

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Documents relating to the following products were received:

Palatinol A.H. (diethyl hexyl phthalate), a plasticizer made from phthalic anhydride and ethyl hexanol. Emulgator 1,000 or Nekal B.X. (sodium salt of dibutylnaphthalene sulfonic acid) made by sulfonating a mixture of butanol and hot-pressed naphthalene to form dibutylnaphthalene - monosulfonic acid and converting the latter to the sodium salt with caustic soda and sodium hypochlorite. It was used as an emulsifier for Buna.

Phthalic anhydride, made by the oxidation of naphthalene with air, using a vanadium catalyst containing potassium sulfate. (Described in CIOS Report Item 22, File XXVIII-29.)

Polystyrol E.F., made by polymerizing styrene in a weak, alkaline soap solution with potassium persulfate as catalyst. The polymer is precipitated from the latex by acidifying with formic acid. It is then filtered, washed, and dried. (Described in CIOS Report, Item 22, File XXVII-6, "Diskontinuierliche Destillation B 17.") This document relates to refining butanol-residue, palatinols, and solvents, using discontinuous distillation. It also contains a section on the continuous production of butylacetate from butanol and acetic acid.

Because of insufficient time, it was impossible to visit the plants in which the products mentioned above were made.

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Capacity, tons per month

	Present position	After repair of bomb damage
Ethyl benzene	2,700	2,700
Styrene	1,600	1,600
Poly-styrene	50	50
Quicklime	15,000	15,000
Hydrogenated ethylene	2,200	2,200
Acetic acid	1,200	1,200
Acetone	200	200
Hexane-triol	300	300
Carbide	26,000	30,000
Acetylene	9,000	9,000
Acetaldehyde	11,000	11,000
Butylene-glycol	11,000	11,000
Butadiene	5,000	5,000
Buna S	4,800	6,000
Emulsifier 1,000	300	300
Numbereo buna	270	270
Ethylene spirits	600	600
Ethylene oxide	350	700
Glycol	700	700
Chlorine	3,000	4,000
Hydrogen chloride	1,600	2,100
Caustic soda	3,400	4,300
Vinyl chloride	3,200	3,200
Igelite PCU	350	600
Ethyl chloride	120	120
Tetrachlorethane	700	700
Trichlorethylene	400	400
SS-oil	500	500
Aluminum chloride	700	700
Formaldehyde	750	750
Butene-diol	500	500
Butane-diol	500	500
Tetrahydrofurane	300	300
Phthalic acid	270	540
Palatinol	150	150

Chemische Werke Albert, Wiesbaden-Biebrich

Alkyd and Phenolic Resins

Chemische Werke Albert, Albertstrasse, Wiesbaden-Biebrich, was visited on September 10. The persons interviewed were Dr. Arthur Greth, manager and chemist for the development of synthotic resins, and Dr. Stiegel, plant chemist. The chief manager of the plant is Dr. Winkler, but he was not interviewed.

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The purposes of this visit were as follows:

1. To find out whether this company, as reported by one investigator, makes phthalic anhydride.

2. To learn whether and how phthalic anhydride or other coal-tar derivatives are used by this firm.

3. To obtain for the TIIC-Forest Products Group, at the request of D. G. Coleman, as much information as possible about the formula, manufacturing procedure, and uses of Albert's glue No. 319-J.

In answer to the first question, Dr. Greth said that his company does not make phthalic anhydride; that all phthalic in Germany is made by I. G. Farbenindustrie, the principal plant being at Ludwigshafen and another at Schkopau.

Concerning item 2, Dr. Greth said that his firm is a large user of phthalic anhydride and phenols. Alkyd resins of both drying and nondrying types are made. Dr. Greth mentioned that whereas he uses adipic acid in some nondrying types, one company in America (Resinous Products Co.) uses sebacic acid.

Dr. Stiegel and Dr. Greth furnished the following information about their alkyd resins:

No. 246 B - Linseed-oil type (air-drying).

Formula:

	Kgs.
Linseed oil	1,930
Phthalic anhydride	1,190
Glycerol	565

Procedure:

The linseed oil and phthalic anhydride are placed in the kettle together and heated to 200°C. The glycerol is then run in, and heating is continued until 240°C. is reached. Acid number and absolute viscosity, in centipoises (determined by Ubbelohde capillary viscometer), are determined.

When the acid number is about 30, vacuum (about 700 mm.) is applied to the kettle and held until the acid number is below 10 and the viscosity is between 35 and 40 for a 1:1 solution of the resin in toluene. The resin is then cooled to 180°C. and the kettle is emptied.

Other grades of this same alkyd having higher viscosities are made according to the same formula, but the vacuum is applied for a longer time.

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No. 125-Z linseed-oil long oil-type alkyd (air drying)

Formula:

	Kgs.
Linseed oil	1,800
Phthalic anhydride	480
Glycerol	225

Procedure:

Same as for No. 246-B. Acid number of product should be below 10° and the viscosity about 20° for a 1:1 solution in toluol.

No. 378-B drying-type alkyd made with synthetic fatty acid

Formula:

	Kgs.
Phthalic anhydride	1,140
Linseed oil	1,270
Synthetic fatty acid 1/	250
Hexanetriol (I. G. Farben.)	850
Pentaerythritol	170

1/ Purchased from Henkel. Consists of the first fraction from the distillation of synthetic fatty acid made from Fischer Tropsch paraffin. Chain length, 4 to 9. Called vorlauf fettsäure in Germany.

Procedure:

All of the ingredients are placed in the kettle together and the mixture is heated to 215°C. It is held at that temperature until the acid number is 35, when the vacuum is applied and held until the acid number is about 15 and the viscosity 30 to 35 centipoises for a 1:1 solution in toluene.

No. 358-D (formerly No. 248 D) air-drying type made with castor oil (medium oil type)

Formula:

	Kgs.
Castor oil (not dehydrated)	2,240
Phthalic anhydride	1,000
Glycerol	440

Procedure:

Put all ingredients in kettle together. Heat as rapidly as possible to 270°C. Apply vacuum (700 mm.). Determine the viscosity. The viscosity

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drops to a minimum, and if heating is continued at the same temperature it will rise again. The minimum is the point at which dehydration is completed. It should be about 15 centipoises (1:1 in toluene). At this point the temperature should be lowered to 240° and heating continued at this temperature until the viscosity is 35 centipoises and the acid number below 10. Cool to 180°C. and empty the kettle.

When resin of this type is to be used in white enamels it is desirable to substitute soya-bean oil for a part of the castor oil.

No. 380-D (formerly No. 198-Z) air-drying-castor oil (long oil) type.

Formula:

	Kgs.
Castor oil	1,860
Phthalic anhydride	615
Glycerol	260

Procedure:

Same as for No. 358-D.

No. 251-G baking type-castor oil (short-oil type).

Formula:

	Kgs.
Castor oil	1,025
Phthalic anhydride	900
Glycerol	500

Procedure:

Same as for No. 358-D, except final viscosity (1:1 in toluene should be 300-400 centipoises and acid number about 20).

The following are some of the nondrying types of alkyds made by Chemische Werke Albert:

No. 166-S medium oil type, nondrying alkyd.

Formula:

	Kgs.
Phthalic anhydride	920
Synthetic fatty acid	1,630
Pentaerythritol	1,020

Procedure:

All ingredients placed in kettle together. Mixtures heated at rate of 15° per hour to 240°C., held at that temperature until acid number is 10-15 and viscosity (1:1 in toluene) 20-30 centipoises. Cooled to 180° and emptied.

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No. 215-S long-oil type, nondrying alkyd

Formula:

	Kgs.
Synthetic fatty acid	2,060
Phthalic anhydride	780
Pentaerythritol	930

Procedure:

Same as for No. 166-S, except the viscosity of final product (1:1 in toluene) should be 8-12 centipoises.

No. 393-S nondrying alkyd, adipic-acid type.

Formula:

	Kgs.
Synthetic fatty acid	1,970
Adipic acid	860
Pentaerythritol	955

Procedure:

Same as for No. 166-S. Final viscosity (1:1 toluene) 80-100 centipoises and acid number 10-15.

No. 398-A nondrying adipic-acid type.

Formula:

	Kgs.
Adipic acid	195
Triethylene glycol	1,500
Glycerol	350

Procedure:

Same as for No. 166-S. Final viscosity (1:1 in butyl acetate) 80-100 centipoises and acid number about 15.

Phenolic Resins

Chemische Werke Albert makes many types of phenolic resins. For some of them it makes a special base from phenol and acetone, which it calls Dian. The reaction is as follows:

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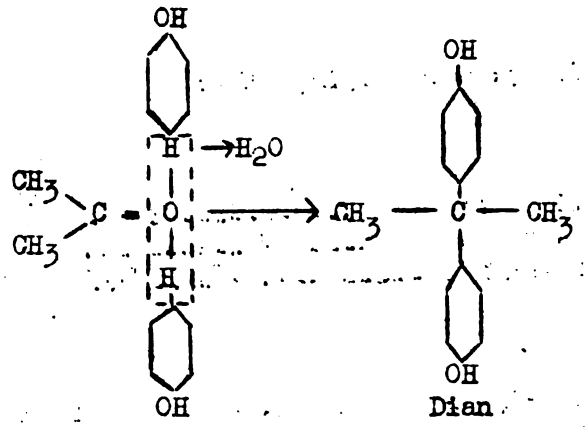

Dian

For "Albertols," modified phenolic resins, diphenylol propane is condensed with formaldehyde. Albertols are described as combinations of phenolic resins with natural rosin acid (abietic acid).

Following is a typical formula for a modified (plasticized) phenolic for varnish purposes as made by Albert.

No. 308-V Modified (plasticized) phenolic

Formula:

	Kgs.
Cresylic acid (35 percent meta and balance para and ortho)	1,080
Formaldehyde (30 percent)	2,000
Ammonia (25 percent)	40
Butanol	800
Tall oil ester (glycerol)(contains about 35 percent abietic acid and balance is fatty acid similar to linseed oil fatty acid)	1,250
Xylene	850

Procedure:

The cresylic acid is placed in the kettle and heated to 60°C. Then a mixture of the formaldehyde and ammonia is run in with cooling. After the formaldehyde-ammonia mixture has all been added the water is distilled off at 50°-55°C. under vacuum (700 mm.). Dilute with the butanol and add the tall oil ester. Then distill off about 50 liters of butanol and water per hour. When a viscosity (1:1 in toluene) of 140 centipoises is reached, dilute with the xylene. The final viscosity should be 20 to 25 centipoises and the solid content 65 percent.

Glue No. 319-J

Information concerning the manufacture of glue No. 319-J, requested by Mr. Coleman, was submitted to B. H. Wilcoxon in a letter dated September 10, 1945. A copy is hereto attached.

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Other Products

A list of the synthetic resins and products based upon resins manufactured by Chemische Werke Albert at Wiesbaden-Biebrich accompanies this report.

Manufacturing Facilities

The resin-making plant on Albertstrasse, Biebrich, was heavily damaged by bombs on September 19, 1944. The Dian plant was completely destroyed, also a building that contained most of the resin kettles. A new building, which was to house the new resin plant with gravity flow of materials, was damaged but not destroyed. Six complete, stainless-steel kettles of 5,000 liters capacity each had been installed in the new building but before the air raid were moved to the Schönebeck plant near Magdeburg, so they are no longer available for use by Albert.

Altogether 12 kettles were destroyed by the raid at Biebrich, but 4 kettles of 5,000 liters capacity are now operating and two 500-liter kettles are making phenol-formaldehyde resins. Also, two kettles of 2,000 liters capacity and some smaller kettles are available for the same purpose.

Before the bombing, "Albert" was making about 1,000 tons of varnish resins per month. Present production is about 250 tons per month, of which phenolics amount to about 100 tons and alkyds 150 tons.

The preferred method of heating resin kettles, as used by Albert, is by means of hot water. Water circulates through heating coils in a furnace and then through a coil welded to the surface of the kettle. Albert has replaced most of its steam-jacketed kettles with water-heated kettles of this type. The builder of the hot-water heating system is Sanesreuther, in Butzbach (Oberhessen), near Friedberg.

"B. H. Wilcoxon
TIIC, Miscellaneous Chemicals Group

"This afternoon, during an interview with Dr. Arthur Greth and Dr. Stiegel of Chemische-Werke Albert, Wiesbaden-Biebrich, I obtained the following information concerning glue No. 319-J. This is one of the plastic glues mentioned in Mr. D. G. Coleman's letter addressed to you and dated September 6, 1945.

Formula for No. 319-J

	Kgs.
Phenol	153
Formaldehyde, 30 percent	326
Caustic soda, 33 percent	3.5
Para toluene sulfonic acid of 50 per cent concentration in water solution	9.6
Softener No. 398 A	62.5
Methanol	37.5

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Manufacturing procedure

"To make glue No. 319-J the following procedure is followed:

"The phenol, formaldehyde, and caustic soda are charged together in a stainless-steel, steam-heated kettle provided with a mechanical agitator. The temperature of the mixture is raised to 90°C., and this temperature is maintained until the mixture becomes turbid. The mixture is then cooled to 60°C. and is neutralized with the para-toluene sulfonic acid solution. The mixture is then allowed to settle and the water layer is decanted. The resin is then dehydrated under approximately 700 mm. vacuum and heating, under vacuum, is continued until the viscosity (when the resin is dissolved 1:1 in ethyl alcohol) is 18 to 20 centipoises. (The viscosity determination is made with an instrument of the Ubelohde, capillary, type.) The resin is then diluted with the methanol, and the 398A softener is added. The 398A softener is prepared as follows:

Preparation of No. 398A softener

Formula:

	Kgs.
Adipic acid	195
Triethylene glycol	1,500
Glycerol	350

Procedure: The three ingredients are mixed together in the kettle and heated at the rate of 15°C. per hour to 240°C. This temperature is maintained until the acid number is about 15 and the viscosity of a 1:1 solution in butyl acetate is between 80 and 100 centipoises.

"Attached is a three-page discussion (in German) of the ways in which glue No. 319-J may be used. Of particular importance is the part stating that just before this glue is to be used it must be mixed cold with a catalyst in the proportion 100 parts 319-J to 10 parts of catalyst. The catalyst is prepared as follows: 50 parts of paratoluene sulfonic acid, 25 parts of methanol and 25 parts of 398A are mixed cold to form a solution, which is the catalyst."

Origin of 319-J

Dr. Greth said that the origin of 319-J was as follows: The glue originally used by the air ministry was Kaurit (a water-soluble urea resin from I. G. Farbenindustrie). Its water and mildew resistance was not sufficiently great, especially in the tropics, so the Air Ministry asked Chemische-Werke Albert to develop a more suitable glue. 319-J was produced, and commercial manufacture was just starting when the plant was bombed on September 19, 1944. According to Dr. Greth, no large quantity of 319-J has been made.

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Another glue used during the war was P-600 made by Dynamit at Troisdorf. It was a water-soluble phenolic. Polystal, another resin glue made by I. G., was produced at Ludwigshafen. Dr. Greth said that he was not able to give the compositions of P-600 or Polystal. The writer will inquire further about Polystal and Kaurit on visits to I. G. plants.

Chemische Werke Albert, Wiesbaden-Biebrich

Albert Glue-resin 319-J

The Albert glue-resin is suited to cold-glueing of any kind in every stage of manufacture and can also be applied to the warm-or-hot process of glueing. 319-J sets very quickly and produces, therefore, a very strong and lasting bond in a short time, which will withstand all strains, is resistant to water and micro-organisms, and insensitive to damp or dry heat.

The Albert glue-resin has been developed through several years of intensive research work and has met official standards and large-scale utilization as well as trials under difficult conditions with complete success.

General

The temperer H-25 belongs with the Albert glue-resin 319-J. Both are fluid and are mixed with one another in the production of finished glues.

In cold dry storage the resin becomes harder and stable for months. An eventual viscosity increase of the glue-resins, which takes place only very slowly, is without influence on the adhesive qualities and purity of the product.

Mixing the Glue

The glue must be mixed in vessels of stoneware, glass, porcelain, enamel, or wood. A vessel and stirrer of metal are useless.

In the production of the finished glue, 100 parts by weight of the resin normally would be mixed with 10 parts of temperer H-25 (i.e.; in the ratio of resin to temperer as 10 to 1) during thorough and vigorous agitation, whereas the original violet-red color changes to bluish. Although the weight of the resin can be obtained exactly by simple weighing, the quantity of temperer can be obtained by measuring in a graduated glass; 10 g H25 correspond to approximately 8.9 cubic centimeters, sp. gr. 1.13. To one kg. of resin, 100 grams, or 89 cubic centimeters, of temperer H-25 is added.

Slight variations over or under are permissible; nevertheless, they should not take place without a specific reason, the resin and temperer having been exactly adjusted and standardized against one another. With proper and careful mixing, the glue is immediately ready for use. It remains workable 3 to 5 hours, according to room temperature, and the layers of glue should be applied consecutively after the first application. The glue is

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usable as long as it is possible to brush it on flexibly. The best working temperature lies between 18° and 22°C. At less than 16°C. the glue sets too slowly; at more than 24°C. it is recommended that the gluepot be put into cold water to prolong the period of use. It is suitable to apply a small quantity of glue frequently and to distribute the fresh application over a smaller working area. For best results, smooth working and dependable strength is decided by glue application and distribution.

Application of the Glue

The finished glue is clear, of low viscosity, and can be applied with pencil, brush, or spatula. The application should be adequate but not excessive. The glue is always applied to both surfaces to be glued.

Dilution of the finished glue is not necessary. For special bonding an entirely special, thinly fluid glue would be prepared by adding 100 parts by weight to not more than 5 to 10 parts of methanol or ethanol. From this, it is apparent that every addition lengthens the setting time. The addition of other organic diluents or solvents is not advisable.

For all glueing work using the thinner application, two applications of the glue are advisable.

After applying the glue, the glued surfaces should remain open (to the air) as much as 30 minutes. Drying in this manner is specifically for treating hard wood, plywood, and layers of wood, and is also necessary when glueing larger surfaces. Scotch pine can be treated under certain conditions without drying.

Large glued surfaces should be freshened again with a spatula, comb, etc., after being allowed to dry suitably. During drying, this portion must not be brought into the vicinity of ovens, heaters, etc., where irregular drying may take place and may cause the glueing to fail.

Pressing and Further Operations

After drying, the single pieces should be joined, even by force, and set under pressure in a pressing device as soon as possible.

Leaving the joined piece lying without pressure is to be avoided. After 3 to 5 hours of pressing, the glue strength becomes so great that the joined piece can be removed from the pressing device. For pieces under tension, as well as for larger surfaces and other difficult jobs, holding for a longer pressing time is desirable; the optimum pressing time can be ascertained easily in such cases through a preliminary experiment. The strength of the glueing also increases after removal of pressure and completely reaches its maximum only after several days. It is therefore advantageous to let the glued piece remain at least 12 to 24 hours before retreating another time. This is particularly true when the glued piece is treated further to achieve required mechanical strength.

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Warm and Hot Glueing

The Albert glue-resin 319-J is also very well suited for warm and hot glueing. The basic principles are the same as those regulating the thorough mixing, application, and drying of cold-glueing process. The amount of temperer, however, can be lowered from one-half to one-quarter of the usual amount for cold-glueing. In general, only 2.5 to 5 parts of temperer H-25 would be added to 100 parts by weight of glue-resin.

The pressing time then declines from 2 hours to 3 minutes, according to the job, the mode of application, and the temperature. Here, also, the optimum pressing time, temperature, and amount of temperer for a suitable series of compositions should be determined by preliminary experiment. The glue is colored dark-red by warm and hot glueing, which usually occurs only after some time.

Glueing Preserved Woods

319-J can be used on woods that, for example, have been preserved with aviation E varnish, just as was shown on a similar 319-J glue layer. For application of other preservatives, corresponding experiments should be made.

Other Directions

Hardened glue residue is no longer usable. It cannot be converted again into usable form with a solvent or other addition. It is best for the still fresh glue residue to be removed from the receptacle at the conclusion of the work. Glue residue that has become hard can be loosened by boiling with an alkali cleansing agent.

An mixture of equal parts of water and methyl or ethyl alcohol serves well for hand cleaning.

Field of Production

I. Work Albertstreet, Amoeneburg.

A. Artifical resins, chiefly used as raw material for the fabrication of varnishes and lacquers; artifical resins for varnishes.

1. Albertols: Modified phenolic resins; combination of phenolic resins with natural rosin acid (abietic acid). Chief field of application: fabrication of oil-varnishes, combination lacquers, cellulose lacquers, spirit varnishes, printing inks, tinctures.

2. Alphenates: Mixed ester of phenolic resins; for oil varnishes and cellulose lacquers.

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3. Alresene: Pure, oil-soluble, phenolic resins; wood-oil-reactive; pure alkylphenolic resins for varnishes with a special resistance against water, acids, and alkalies.

4. Alnovols: Pure, spirit-soluble, phenolic resins, for spirit varnishes, polishes, matting varnishes, adhesive substances.

5. Phenodure: Pure, spirit-soluble, hardening, phenolic resins; binding agent for different purposes, e.g., for the production of grinding wheels, brake linings, and carbon brushes.

6. Durophene: Plastified, thermo-setting phenolic-resins for stoving varnishes, enamels, electro-insulating varnishes, nitrocellulose lacquers.

7. Alresate: Maleic acid resins; pale, artifical resins, free of phenol, with excellent light and color resistance; for oil varnishes and cellulose- and combination lacquers.

8. Alftalats: Alkyd resins, i.e., mixed esters of dicarbonic acids (e.g., phthalic acid) and fatty acids; for air-drying and stoving varnishes of every kind, cellulose lacquers, combination lacquers, enamels, often in combination with Albertols and Alresates.

9. Resamine: Urea resins for varnishes and enamels, possessing high heat resistance; combination with alkyd resins and nitrocellulose; binding agent for stoving enamels possessing a high gloss.

A branch factory for the types of artifical resins mentioned above exists at Schönebeck/Elbe.

B. Special products based upon artifical resins:

1. Rosin size for the paper industry, based upon natural rosin and artifical resin;

Universal auxiliary agent for the fabrication of writing, packing, and similar papers to make them waterproof and to give them mechanical stability and smoothness; especially impregnating media for special papers with higher water resistance.

2. Glue for wood based upon phenolic resins:

Glues for wood for the hot and cold process, obtaining a special water- heat- and tropical resistance; for the production of plywood, hardwood and for glueing purposes for aeroplane construction, wooden carriage bodies, boat building, furniture industries.

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Carbon-Black Production at Dortmund-Ruhr

Deutsche Gasrusswerke G.m.b.H.

(Average conditions in 1942 for production of 1 kilogram of product)

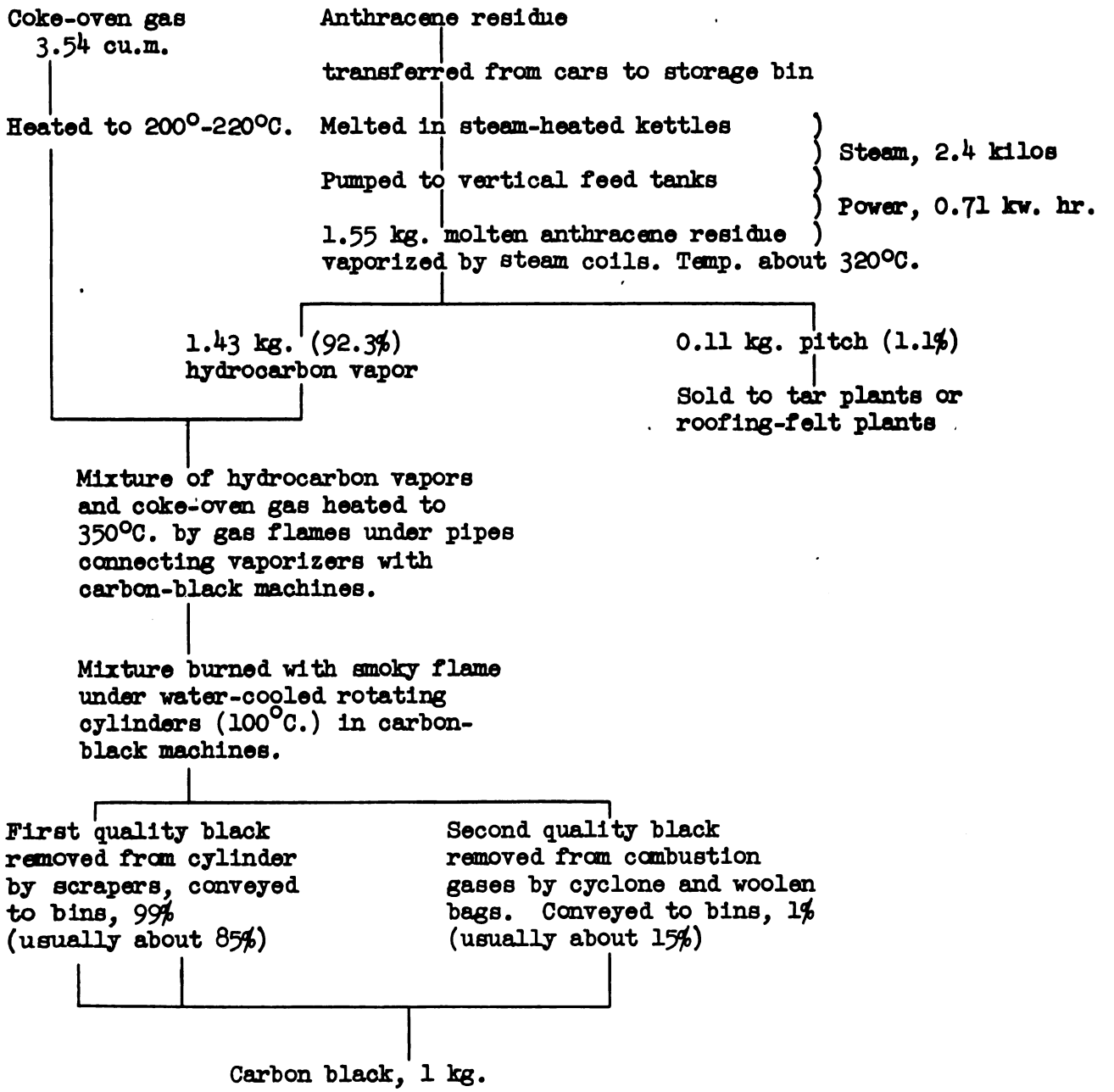
Coke-oven gas
3.54 cu.m.
Heated to 200°-220°C.
Anthracene residue
transferred from cars to storage bin
Melted in steam-heated kettles
Pumped to vertical feed tanks
1.55 kg. molten anthracene residue vaporized by steam coils. Temp. about 320°C.
Steam, 2.4 kilos
Power, 0.71 kw. hr.
1.43 kg. (92.3%) hydrocarbon vapor
0.11 kg. pitch (1.1%)
Sold to tar plants or roofing-felt plants
Mixture of hydrocarbon vapors and coke-oven gas heated to 350°C. by gas flames under pipes connecting vaporizers with carbon-black machines.
Mixture burned with smoky flame under water-cooled rotating cylinders (100°C.) in carbon-black machines.
First quality black removed from cylinder by scrapers, conveyed to bins, 99% (usually about 85%)
Second quality black removed from combustion gases by cyclone and woolen bags. Conveyed to bins, 1% (usually about 15%)
Carbon black, 1 kg.

Figure 8.

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3. Emulsions for textiles based upon artifical resins:

To impregnate yarns and fabrics, especially made of artifical silk, in order to improve firmness when washing; to render it wind-proof and resisting against putrefaction; further to reduce shrinking of the material.

C. Moulding powders:

Moulding powders are a combination of hardening artifical resins and fillers of different kinds. They are pressed in press moulds under the influence of heat and pressure to obtain the pressed articles.

1. Alberit-Quick-pressing moulding powder, type 1: Asbestos fiber used as filler; this moulding powder enables preparation of high-heat-resisting insulating material.

2. Alberit-Quick-pressing moulding powder, type S: Wood powder used as filler; this moulding powder is used for the production of articles for daily use and for electrical insulating material.

3. Alberit-Quick-pressing moulding powder, type T: Shavings and layers of textiles used as filler; apart from insulating properties high mechanical resistance.

4. Alberit-Quick-pressing moulding powder, type Z: Cellulose used as filler; high mechanical resistance and insulating properties.

Deutsche Gasrusswerke G.m.b.H., Dortmund

Two visits were made to this plant - on September 21 and November 9, 1945. The persons interviewed were Dir. Bache und Dr. Herrmann. Damage to this plant by air raids was confined principally to the office building.

Anthracene residue in the form of a coarse powder is stored in large bins, from which it is moved by an overhead crane to melting kettles equipped with mechanical stirrers. From the melters it is pumped to feed tanks, from which the molten anthracene residue flows into gas-heated vaporizers, where the vapors of the residue are combined with preheated coke-oven gas. The mixture of gas and vapors is circulated through pipes heated by gas flames to the machines that make the carbon black. In each machine is a rotating, water-cooled roller, beneath which is a row of burners for the gas-vapor mixture. Incomplete combustion of the mixture at the burner tips causes carbon black to be deposited on the roller. It is removed by a doctor blade and conveyed to a storage and bagging center. The process is shown diagrammatically in the accompanying flow diagram (fig. 8).

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Peak production at this plant was reached in 1942, when 90.1 percent of its capacity was used. The quantities of carbon black made were 13,897 tons first quality and 166 tons second quality - total, 14,063 tons. The quantities of raw materials used were 49,783,000 cubic meters coke-oven gas, 21,798 tons (metric) of anthracene residue, and 33,614 tons of steam. The equivalent quantities per kilogram of carbon black are 3.54 cubic meters coke-oven gas, 1.55 kilograms anthracene residue, and 2.4 kilograms steam. Power consumption was 0.71 kw. hr. per kilogram of carbon black. The yield of carbon black is 72.5 percent when referred to anthracene residue actually vaporized, or it is 64.4 percent of the original anthracene residue used. The remainder is pitch residue (7.7 percent) and combustion loss (28 percent).

Prices paid in 1942 for raw materials were: anthracene residue, 5.40 RM per 100 kilograms, and coke-oven gas, 0.015 RM per cubic meter. The cost of the finished carbon black was 0.75 RM per kilogram.

Anthracene oil can be used satisfactorily at the Dortmund plant but is much more expensive (12.00 RM per 100 kilograms). Naphthalene is a satisfactory raw material but, in addition to being more expensive (9.00 RM per 100 kilograms for 76-degree naphthalene and 11.50 RM for hot-pressed), some changes would have to be made in equipment and operating procedures at Dortmund to use naphthalene there. The Kalscheuren plant at Cologne, having 25 percent the capacity of the Dortmund plant, can use naphthalene and anthracene residue interchangeably.

Plants that produce lamp black by partial combustion of anthracene oil are located at Worms, Zons, and elsewhere.

Ruhröl G.m.b.H., Bottrop-Welheim

The Bottrop-Welheim hydrogenation plant of Ruhröl G.m.b.H. was visited on September 21, 1945. The persons interviewed were Direktor Dr. Frese and Dr. Broche. This plant was of particular interest because of

1. Its use of a mixture of 70 percent briquette pitch and 30 percent coal-tar oil as the feed stock for the hydrogenation plant.

2. The digestion of coal in medium oil from the hydrogenation process, followed by filtration through ceramic filters to obtain a coal extract.

3. The vacuum-distillation of the coal extract to obtain a high melting-point pitch suitable for charging directly into coke ovens to make low-ash coke for carbon electrodes.

Also, a residue from liquid-phase hydrogenation (Entschlammung) went to the tar-distilling plant of the same organization for distillation to briquette pitch in admixture with coal tar.

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The hydrogenation of the pitch-tar oil mixture, as it was carried out at this plant, has been discussed in detail by C. Cockram in CIOS Report Item 30, File XXX-104.

The coal-extraction and filtration process also was discussed by Cockram in the CIOS report mentioned above and by Lowry and Rose in CIOS Report Item 30. Additional information about this type of filter is presented in a separate report by this investigator, covering a visit to Schumachersche Fabrik in Bietingheim where the ceramic filter rings are made. The following description of the filtering operation was furnished by Dr. Frese:

> In a pressure vessel are mounted vertical tubes composed of porous stone rings, the so-called candles or cartridges. The liquid pumped into this vessel flows around the outsides of the candles. An absolute pressure of 4 to 5 atmospheres is gradually applied to the mixture, causing the liquid portion to penetrate into the interior of the tubes, from which it is drawn off at the bottom, while the solid components of the mixture deposit on the exterior surfaces of the candles. When the filter cake has grown to a certain thickness, the raw product is poured back from the filter cake into the raw-product feed tanks by means of CO_2. The filter case is then filled with washing filtrate, pure filtrate or solvent, depending on the state of the product and is forced through the filter cake into the candle, so that the residual carbon cake finally contains only solvent. After emptying the filter case, the cake is dried by hot CO_2. Beneath the filter case is a conical funnel sealed by a patented closure. The latter is opened, and CO_2 is introduced into the interior of the candles under an absolute pressure of 8 atmospheres. By this means the cakes are pushed off the filter rings. They fall down into the so-called "protecting gas tank" from which the escaping CO_2 is conducted into the open air through pipes terminating on the roof. The lower part of the protecting gas tank is provided with a discharging device which carries the filter cake into a chain conveyer below. In the gas-tight compartment of this conveyer the residue coal is transferred to building 13a where the solvent which it contains is eliminated.

Each of the three filters used for the above-described operation consisted of a steel case with cone bottom, inside of which were 27 filter tubes or candles. Each candle was made up of 68 porous ceramic rings clamped together to form a porous tube 2,050 mm. long, 120 mm. O.D., and 80 mm. I.D. (each ring was 20 mm. high). After about 5,000 filtrations of 15 minutes each, the candles were removed from the filter, and new rings were substituted for those that had been in use.

The filtered extract was distilled in a special plant consisting of a gas-heated pipe still and two gas-heated batch stills with mechanical agitators. The filtered extract was pumped through the pipe still and two gas-heated batch stills with mechanical agitators. The filtered extract was

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pumped through the pipe still into a batch still, where the vapors were released and the distillation was finished. The distillation residue was discharged to a continuous steel-plate cooling conveyor, which transferred the cooled pitch into cars.

The distillation of the residue from the Welheim hydrogenation plant in combination with coal tar at the tar plant of Mathias Stinnes is discussed by this investigator in a separate report.

Ruhrchemie A.G., Sterkrade-Holten

The primary purpose of a visit to Ruhrchemie A.G., Sterkrade-Holten, on September 19, 1945, was to find out what coal-tar products, if any, were used by that company, the investigator having heard that naphthalene had been employed in connection with the Fischer-Tropsch synthesis.

Director Martin, when interrogated about this matter, stated that some naphthalene from tar plants had been used with olefins from the Fischer-Tropsch synthesis in the making of lubricating oils by the aluminum chloride process. However, he stated that lubricating oil made from a mixture of naphthalene and Fischer-Tropsch olefins was inferior to that from Fischer-Tropsch olefins alone because of poorer thermal characteristics.

(Note: At a later date it was learned that the use of naphthalene in lubricating oil in the manner mentioned above was practiced at the Fischer-Tropsch plant of Rheinpreussen at Homberg-Moers instead of at Sterkrade-Holten. This has been reported by investigators Atwell and Schroeder in CIOS Report Item 30, File XXIV-9. According to that report, Fischer-Tropsch medium oil, boiling range 250°-350°C., was chlorinate at 80°-100°C. to 20 to 25 percent chlorine by weight. Five volumes of the chlorinated oil were reacted with two volumes of naphthalene at 70°-100°C. in the presence of eight volumes of Fischer-Tropsch benzine and a small amount of $AlCl_3$ as catalyst. After withdrawal of sludge, neutralization with lime, bleaching, filtering, and removal of benzine, the product was vacuum-distilled to make spindle oil, turbine oil, and cylinder stock. According to Atwell and Schroeder "all products were stated to have a high viscosity index and great resistance to oxidation, but tests on captured samples do not support these claims.")

In further conversation with Dr. Martin, he mentioned the use of "vorlauf fettsäure" (produced at soap factories from Fischer-Tropsch paraffins) with phthalic anhydride from naphthalene in the making of alkyd resins. He said that these were the only uses, as far as he knew, for coal-tar products in connection with Fischer-Tropsch products. No use of tar products was contemplated in connection with the operation of the Oxo process.

Following the discussion outlined above, a brief visit was paid to the Fischer-Tropsch and Oxo plants. In the Fischer-Tropsch plant, the buildings were badly damaged or destroyed; but, according to Dr. Martin, at least 50 percent of the equipment can be used again. The Oxo plant was damaged only

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slightly. No detailed inspection of the Fischer-Tropsch and Oxo plants was made, because they have been covered by previous investigators.

Gasinstitute der Technische Hochschule, Karlsruhe

On December 1, 1945, a brief visit was paid to the Gasinstitute der Technische Hochschule, Karlsruhe, Germany, den Schlacthausstrasse 3, for the purpose of determining whether this research institution had carried on any investigational work in recent years on coal tar or products derived therefrom.

The persons interviewed were Professor Köerting and Professor Weyrich. Professor Bunte, former director of the Gasinstitute and well-known authority on gas purification, gas production, etc., died last year. Professor Köerting explained that previously the work of the Gasinstitute was of two types. One phase of its activities was the teaching of gas utilization in the Technische Hochschule. This department was conducted by Professors Bunte and Köerting. Professor Köerting hopes that this work will be resumed soon by permission of the Military Government.

The other department or section of the Gasinstitute, according to Professor Köerting, resembled the American Gas Association. It dealt with research on and the practical testing of gas and water and was financed by the gas industry.

The Gasinstitute was housed in a separate building within the plant of the Statische Werke, Karlsruhe Gaswerk, Ost. This building was almost totally destroyed, but a complete experimental gas plant consisting of two horizontal retorts and all the auxiliary equipment was undamaged. Also, the analytical laboratories in the basement were not destroyed. Dr. Köerting hopes to be able to use these facilities for further research work if funds for such work can be obtained.

At present, Professor Köerting, Professor Weyrich, and an assistant are occupying a small room in the office and laboratory building of the gas plant and are unable to carry on any of their usual activities.

When questioned about work on tar or tar products, neither Professor Köerting nor Professor Weyrich could remember having done anything along this line within the last 10 years.

The Didier Werke inclined chamber ovens at this gas plant were visited with Professor Köerting, but no description of them will be given here because they are the subject of final FIAT Report No. 566.

Kaiser Wilhelm Institute für Kohlenforschung, Mülheim, Ruhr

This research laboratory was visited on November 21, 1945, with H. P. Stephenson, of North German Coal Control, and Major Henri Winkler. Neither the building nor any of the laboratory equipment had been damaged during

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the war, and research is being resumed, with permission of the Military Government, on the production of higher paraffins by Fischer-Tropsch synthesis methods. The apparatus used for this work was inspected briefly, as well as laboratory fractional distillation equipment. Of particular interest in the latter connection were the rotating band columns, which were said to be highly effective fractionating devices, and, because of low hold-up, they were considered to be especially suitable for the distillation of small samples. A description of this type of column by H. Koch, F. Hilberath, and F. Weinrotter was published in Die Chemische Fabrik, vol. 14, 1941, page 387, under the title "Eine Kolonne mit Rotierendem Metallband Zur Fraktionierten Destillation kleiner Substanzmengen." A copy of the article was obtained.

Dr. Koch, Dr. Peckler, and Dr. Siegler were asked if any research work on coal-tar products had been done in their institution in recent years or in the laboratories of other research institutions in Germany. They were of the opinion that little or no work of that kind had been done outside the laboratories of the larger tar-distilling companies and did not know how much had been done there.

APPENDICES

Quantities of Crude Tar Processed by German Tar Distillers in 1941, 1942, and 1943

Tar-distilling company and plant	Crude tar processed (metric tons) 1941	1942	1943
Western tar plants			
Gesellschaft für Teerverwertung			
(2) Duisberg-Meiderich	772,130	775,385	292,887
Alsdorf			39,872
(2) Rauxel			473,978
(1) Bochum-Gerthe	27,773	25,888	22,666
Rütgerswerke A.G.			
(2) Rauxel	219,610	224,623	172,247
Duisberg			61,801
Gelsenkirchener Bergwerks A.G.			
(2) Bochum (Carolinenglück)			113,324
(2) Wanne Eickel (Pluto)	175,234	188,643	29,113
Duisburg-Hamborn (Thyssen)			4,352
Dortmund (Horder Hüttenverein)			8,023
(1) Bergbau A.G., Ewald König Ludwig, Herten	43,500	50,500	58,668
(2) Teerdestillation Math Stinnes Essen-Karnap	38,634	38,482	39,333
Harpener Bergbau A.G., Dortmund	4,260	146	97
(3) Gebr. Stumm G.m.b.H., Essen-Borbeck	18,695	19,435	4,192

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Tar-distilling company and plant	Crude tar processed (metric tons) 1941	1942	1943
(2) Gutehoffnungshütte Oberhausen-Sterkrade	72,674	72,656	73,556
Steinkohlenberg w. Rheinpreussen, Homberg	28,914	27,765	24,593
(1) Dr. F. Raschig G.m.b.H. Langendreer	38,505	38,388	34,833
Preuss Bergwerks u Hütten A.G., Barsinghausen	5,425	5,427	4,723
G. B. Ranke G.m.b.H., Dortmund	5,693	5,658	5,598
Totals for western plants	1,451,038	1,473,196	1,463,856
Northern tar plants			
(2) Hanseatische Teerproducten Fabrik, Hamburg	31,636	32,381	34,995
(1) Norddeutsche Kohlen u. Cokeswerke, Hamburg	7,280	6,734	4,007
Hochofenwerk Lübeck A.G., Lübeck	9,776	4,068	
(2) Akt. Ges. Johannes Jeserich, Hamburg-Eidelstedt	3,564	3,025	2,584
Stadtwerke Kiel, Kiel	2,833	3,693	4,939
Totals for northern plants	55,089	49,901	46,525
Southern tar plants			
Chem. Fabrik Weyl, Mannheim-Waldhof (2)	69,447	67,353	72,514
Chem. Fabrik Weyl, München-Pasing	17,302	17,521	19,474
Dr. F. Raschig G.m.b.H. Chem. Fab. Ludwigshafen (2)	46,595	49,849	47,493
Teerdestillation Stuttgart G.m.b.H., Stuttgart (1)	20,730	18,620	15,140
Oberrheinische Ölindustrie J. Glaser, Freiburg	6,111	6,557	8,635
Süddeutsche Teerindustrie Aug. Peters, Malsch	1,910	1,712	2,171
Chem. Fab. Badenia, Phil. Keilmann, Mannheim	8,020	9,445	9,000
Chem. Werke Worms Weinsheim G.m.b.H. Worms	7,441	7,355	7,411
Teerdestillation Nürnberg, Nürnberg	5,086	6,994	5,585
Totals for southern plants	182,642	185,406	187,423
Saar tar plants			
Neunkircher Eisenwerk A.G., Neunkircher	27,862	26,847	27,683
Röchlingsche Eisen u. Stahlwerke, Völklingen	38,527	33,797	46,937
Halbergerhütte G.m.b.H., Brebach	15,460	16,440	15,360
Saargruben A.G., Kok u. Chem. Werke, Saarbrücken	3,465	7,037	9,364

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Tar-distilling company and plant	Crude tar processed (metric tons) 1941	1942	1943
Ernst Hugo Sarg & Co., Saarbrücken	10,579	9,916	14,040
Eisen u. Stahlwerk "Carlshütte", Diedenhofen	3,128		
Hüttenver Westmark G.m.b.H., Hagendingen..	11,041	11,913	22,600
Teerdestillation Gaudach, Gaudach	8,508	16,579	10,831
Schook-Streng Nachf. Kg. Teerwerk, Esch ..	3,736	3,237	6,541
Totals for Saar plants	122,306	125,766	143,455
Eastern tar plants			
Rütgerswerke A.G.			
(2) Erkner (near Berlin)	58,545	59,804	66,892
(3) Breslau	42,492	46,284	46,798
(4) Niederau	52,117	47,601	42,943
(5) Teerag A.G., Wien	30,325	31,694	28,153
(5) Borsig Kokswerke A,G., Hindenburg ...	66,591	65,133	59,884
(5) Schaffgotsch Bergswerks ges. m.b.H., Gleiwitz	25,512	25,909	26,709
(5) Oberhütten, ver. Oberschles Hüttenwerke, Gleiwitz	18,916	24,272	28,461
(5) Kokerei Vereinigung G.m.b.H. Kattowitz	82,182	76,634	65,375
(5) Bergwerksverwaltung Oberschles, Kattowitz	36,547	34,435	35,261
(1) Gewerkschaft Morgenstern, Zwickau ...	7,208	7,132	6,536
(1) Döbelner Chem. Fab. Oswald Greiner, Döbeln	8,398	8,282	8,424
(1) Chem. u. Teerproducten Fab. Schwieck & Co., Velten	9,820	10,550	9,670
(5) Imbusch u. Melzer, Waldenburg	4,584	4,649	4,592
(5) Küstriner Teerwerke, Küstrin-Neustadt	2,760	2,620	3,150
(5) Teerindustrie A.G., Danzig	9,915	8,615	9,550
(5) Teerwerk Preussen G.m.b.H., Königsberg	2,888*	5,753	5,518
Totals for eastern plants	455,912	459,367	447,496
Totals for all plants	2,267,017	2,293,636	2,288,728

(*) After June 1941.
(1) Intact.
(2) Damaged.
(3) Destroyed.
(4) Removed.
(5) No longer German.

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Origins of Crude-Tar Supplies Received by the Principal Tar-Distilling Plants of Germany (in 1942/43)

West:	
Gesellschaft für Teerverwertung:	
Works Meiderich	Almost exclusively from coke ovens of the mines in the Ruhr district. Minor parts also from steel works coke ovens.
Works Rauxel	100 percent from coke ovens of the mines in the Rhine-Ruhr area.
Works Alsdorf	100 percent from coke ovens of mines in the Aachen area.
Works Bochum-Gerthe	Only from coke ovens of Bergbau A.G. Lothringen.
Rütgerswerke:	
Works Rauxel	From coke ovens of mines in the Rhine-Ruhr area.
Works Hochfeld	From coke ovens of mines in the Rhine-Ruhr area; small parts from gas works in the Rhine province.
Gelsenkirchener Bergwerks AG:	
Works Carolinenglück, Bochum	Only from coke ovens of mines belonging to the concern.
Works Pluto Wilhelm, W-Eickel	Do.
Ewald-König Ludwig, Herten	Do.
Gutehoffnungshütte Oberhausen	Do.
Gew. Mathias Stinnes, Essen-Karnap	Do.
Rheinpreussen, Homberg	Do.
Gebr. Stumm, Essen-Borbeck	Only from coke ovens of mines belonging to the concern.
Dr. F. Raschig, Bo-Langendreer	Chiefly from coke ovens of the mines in the Rhine-Ruhr area; smaller quantities also from steel-works coke ovens and unimportant quantities from gas works.

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E a s t:	
Rütgerswerke:	
Works Erkner	Main source municipal gas works Berlin; unimportant quantities from gas works of the Province of Brandenburg.
Works Mochbern	From coke ovens of the mines in lower Silesia; besides this, from municipal gas works of Breslau.
Works Niederau	From the gas works of city of Dresden and cities in the vicinity.
"Teerag" Vienna	From the municipal gas works, Vienna.
Emmagrube, Kattowitz	Only from coke ovens belonging to the concern.
Kokerei-Vereinigung, Kattowitz	From coke ovens of the mines in western and eastern Silesia.
Borsig-Kokswerke, Hindenburg	Do.
Oberhütten, Gleiwitz	Only from coke ovens belonging to the concern.
Schaffgotsch, Oderthal	Do.
S o u t h:	
Weyl Mannheim	Main source coke ovens of mines and steelworks in the Saar area. Smaller parts also from municipal gasworks Mannheim and cities in the vicinity.
Weyl Pasing	From gasworks of city of Munich and cities in the vicinity.
Röchling'sche Eisen-u. Stahlwerke, Völklingen	Only from coke ovens of own steelworks.
Neunkircher Eisenwerk	Do.
Halbergerhütte, Brebach	Do.
Saargruben, Saarbrücken	From own coke ovens of the mines.

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South: (cont'd.)	
Dr. F. Raschig, Ludwigshafen	From coke ovens of mines and steelworks in the Saar area; besides this, smaller quantities from municipal gasworks, Ludwigshafen and cities in the vicinity.
Teerdestillation Stuttgart	From municipal gasworks, city of Stuttgart; besides this, smaller additional purchases from the Saar area.
North:	
Hanseatische Teerprodukten-Fabrik Haltermann, Hamburg	Main source, municipal gasworks, city of Hamburg; smaller quantities from gasworks in the vicinity and from Bremen steelworks.

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